Spalling resistance of fiber embedded ultra-high performance concrete

Zhang, Dong

2020

Zhang, D. (2020). Spalling resistance of fiber embedded ultra-high performance concrete.
Doctoral thesis, Nanyang Technological University, Singapore.

<https://hdl.handle.net/10356/137230>

<https://doi.org/10.32657/10356/137230>

This work is licensed under a Creative Commons Attribution-NonCommercial 4.0
International License (CC BY-NC 4.0).

Downloaded on 26 Apr 2023 09:06:11 SGT

**NANYANG
TECHNOLOGICAL
UNIVERSITY**

SINGAPORE

**SPALLING RESISTANCE OF FIBER EMBEDDED
ULTRA-HIGH PERFORMANCE CONCRETE**

**ZHANG DONG
SCHOOL OF CIVIL AND ENVIRONMENTAL ENGINEERING**

2020

**SPALLING RESISTANCE OF FIBER EMBEDDED
ULTRA-HIGH PERFORMANCE CONCRETE**

ZHANG DONG

School of Civil and Environmental Engineering

A thesis submitted to the Nanyang Technological University
in partial fulfilment of the requirement for the degree of
Doctor of Philosophy

2020

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research, is free of plagiarised materials, and has not been submitted for a higher degree to any other University or Institution.

2019/08/19

.....

Date

.....

ZHANG DONG

Supervisor Declaration Statement

I have reviewed the content and presentation style of this thesis and declare it is free of plagiarism and of sufficient grammatical clarity to be examined. To the best of my knowledge, the research and writing are those of the candidate except as acknowledged in the Author Attribution Statement. I confirm that the investigations were conducted in accord with the ethics policies and integrity standards of Nanyang Technological University and that the research data are presented honestly and without prejudice.

2019/08/19

.....

Date

.....

TAN KANG HAI

Authorship Attribution Statement

This thesis contains material from 2 papers published in the following peer-reviewed journal(s) / from papers accepted at conferences in which I am listed as an author.

Chapter 3 is published as (1) D. Zhang, A. Dasari, and K.H. Tan, On the mechanism of prevention of explosive spalling in ultra-high performance concrete with polymer fibers, *Cement and Concrete Research* **113**, 169-177 (2018). DOI: <https://doi.org/10.1016/j.cemconres.2018.08.012>. (2) D. Zhang, K.H. Tan, A. Dasari, Role of polymer fibers in prevention of explosive spalling in ultra-high performance concrete, in: Ali Nadjai, Faris Ali, Jean-Marc Franssen, O. Vassart (Eds.) 10th International Conference on Structures in Fire SiF'18Belfast, United Kingdom, 2018, pp. 85-91.

The contributions of the co-authors are as follows:

- Prof Tan Kang Hai and Associate Prof Aravind Dasari provided the initial project direction.
- I prepared the manuscript drafts. The manuscript was revised by Prof Tan Kang Hai and Associate Prof Aravind Dasari.
- I performed all the sample preparations and experimental works at the School of Civil and Environmental Engineering and School of Materials Science and Engineering. I also analyzed the data.
- All microscopy was conducted by me in the Facility for Analysis, Characterization, Testing and Simulation. I also did the image analysis.
- Prof Tan Kang Hai and Associate Prof Aravind Dasari provided guidance in the

interpretation of the test results.

2019/08/19

.....

Date

.....

ZHANG DONG

ACKNOWLEDGEMENTS

First of all, the author would like to express his greatest gratitude to his supervisors, Professor Tan Kang Hai and Associate Professor Aravind Dasari, for their invaluable guidance, advice, and encouragement throughout these four years of his Ph.D. program. The author would like to extend his gratitude to JTC corporation for the financial support.

The author owes his deepest gratitude to Dr. Li Ye, Dr. Zhang Yao and Dr. Liu Jincheng for their assistance and valuable recommendations. Appreciation also goes to Dr. Zope Indraneel Suhas, Mr. Weng Yiwei, Mr. He Shan and Mr. Ng Yan Hao for their advice and help on the data analysis, sample preparation and experiments.

Special thanks are also extended to all the technical staffs in Protective Engineering and Construction Technology Laboratories, in particular, Mr. Chelladurai Subasanran, Mr. Tui Cheng Hoon, and Mr. Jee Kim Tian for their kind help in experimental work.

The author also wishes to thank all of members in his group, i.e. Dr. Guan Lingwei, Dr. Kang Shaobo, Dr. Liu Junxian, Dr. Nguyen Minh Phuong, Dr. Zhang Zhongwen, Dr. Wen Jian, Dr. Namyo Salim Lim, Dr. Pham Anh Tuan, Dr. Fu Qiuni, Dr. Chen Kang, Mr. Fan Shengxin, Ms. Nguyen Thi Nguyet Hang, Mr. Wang Su, Mr. Du Panwei, Mr. Ma Youxin, for their comments and helpful discussions.

Finally, the author would like to express his utmost gratitude to his parents, parents-in-law and wife, Li Chen, who always stand by him and support unconditionally.

TABLE OF CONTENTS

Statement of Originality.....	i
Supervisor Declaration Statement.....	iii
Authorship Attribution Statement	v
ACKNOWLEDGEMENTS	vii
TABLE OF CONTENTS	ix
ABSTRACT	xiii
LIST OF PUBLICATIONS.....	xv
LIST OF TABLES	xvii
LIST OF FIGURES	xix
LIST OF ABBREVIATIONS.....	xxv
CHAPTER 1 INTRODUCTION	1
1.1 Research background.....	1
1.2 Scope, methodology, and novelty of research.....	4
1.3 Layout of the report	6
CHAPTER 2 LITERATURE REVIEW	9
2.1 Introduction.....	9
2.2 Development of UHPC	9
2.3 Physiochemical change, microstructure and mechanical properties of UHPC at high temperature.....	10
2.4 Spalling of concrete	13
2.4.1 Definition and types of spalling.....	13
2.4.2 Mechanisms of explosive spalling.....	14
2.4.3 Combined pore pressure and thermal stress spalling.....	22
2.5 Spalling prevention	24
2.5.1 Utilization of protective lining and thermal barrier	24

2.5.2	Utilization of steel fibers	25
2.5.3	Utilization of polymer fibers	27
2.6	Mechanism of spalling prevention with polymer fibers	33
2.6.1	Overview	33
2.6.2	Properties of PP fibers	33
2.6.3	Action of PP fibers in concrete at high temperature	35
2.7	Summary	44
CHAPTER 3 MECHANISM OF SPALLING PREVENTION IN UHPC		47
3.1	Overview	47
3.2	Design of experiment	48
3.2.1	Material and sample preparation	48
3.2.2	Thermal analysis	49
3.2.3	X-ray diffraction	50
3.2.4	SEM observation	50
3.2.5	Gas permeability measurement	52
3.2.6	Spalling test	52
3.3	Result and discussion	53
3.3.1	Spalling behavior and the extent of gas permeability	53
3.3.2	Dimensional changes with temperature	58
3.3.3	Microscopic investigation of the role of PP fibers	60
3.4	Summary	67
CHAPTER 4 SPATIAL DISTRIBUTION OF PP FIBERS IN UHPC		69
4.1	Overview	69
4.2	Material and experimental procedure	70
4.3	Material properties	71
4.4	X-ray CT measurement	71
4.4.1	Acquisition of CT images	71

4.4.2	Imaging processing	72
4.4.3	Image analysis.....	74
4.5	Result and discussion.....	75
4.5.1	Spalling test and permeability measurement	75
4.5.2	Fiber distribution.....	78
4.5.3	Air bubble content and mechanical properties.....	87
4.6	Summary.....	90
CHAPTER 5 CRITICAL FIBER DOSAGE FOR SPALLING PREVENTION...		93
5.1	Overview.....	93
5.2	Theoretical model based on Kozeny-Carman equation	93
5.3	Percolation model	99
5.4	Experimental study	102
5.4.1	Material and specimen preparation.....	102
5.4.2	Determination of parameters in the model.....	103
5.5	Results and discussion	104
5.6	Summary.....	113
CHAPTER 6 EFFECTS OF JUTE FIBERS ON FIRE RESISTANCE OF UHPC		
		115
6.1	Overview.....	115
6.2	Design of experiments	115
6.2.1	Mix proportion and sample preparation.....	115
6.2.2	Compressive strength and mass loss.....	117
6.2.3	Flexural strength	117
6.2.4	Gas permeability test.....	118
6.2.5	Spalling tests	118
6.2.6	Accelerated weathering test	118
6.2.7	Thermal analysis	119

6.2.8 SEM and X-ray computed tomography (X-ray CT).....	119
6.3 Results and discussion.....	119
6.3.1 Mass loss	120
6.3.2 Failure pattern after compressive test.....	121
6.3.3 Compressive strength	122
6.3.4 Flexural strength.....	125
6.3.5 Spalling behavior.....	126
6.3.6 Effect of accelerated weathering curing	131
6.4 Summary.....	133
CHAPTER 7 CONCLUSIONS AND FUTURE WORK.....	135
7.1 Conclusions	135
7.2 Future work	138
REFERENCES	141
APPENDIX A EFFECT OF VARIOUS POLYMER FIBERS ON PREVENTING SPALLING	157
A.1 Mix proportion and experiments	157
A.2 Results and discussion.....	158

ABSTRACT

Ultra-high performance concrete (UHPC), due to its dense microstructure, is highly susceptible to explosive spalling when exposed to fire, which limits its applications in building and construction sectors. It is believed that thermal stress and high vapor pressure are two main causes of spalling. To prevent spalling, one of the most widely accepted methods is addition of polymer fibers that are supposed to relieve vapor pressure by creating paths in concrete at high temperature. Although different theories have been proposed to explain the role of polymer fibers in enhancing gas permeability and reducing explosive spalling, the mechanism is still not fully understood. Moreover, there is not an appropriate method regarding optimizing fiber geometry and fiber dosage on spalling prevention.

In this work, a range of analytical and microscopic characterizations were conducted to thoroughly understand the exact role of polymer fibers in the prevention of explosive spalling of UHPC. Furthermore, to better understand the interconnectivity of fibers and its effect on spalling prevention, X-ray computed tomography (CT) is employed to obtain spatial distribution of polypropylene (PP) fibers in UHPC. The connectivity of PP fibers (and thereby, percolation of fiber clusters) is calculated based on the three dimensional (3D) reconstructed model from X-ray CT results. Spalling tests and permeability measurements are also conducted on UHPC samples with different sizes of PP fibers to reaffirm the mechanism. The results suggest that thermal mismatch between embedded fibers and matrix is critical to obtaining an interconnected network of cracks in the matrix. This occurs even before melting of polymer fibers. The network of cracks is responsible for enhancing permeability, thereby reducing the susceptibility of explosive spalling of UHPC. High fiber connectivity and small inter-fiber distance are required to obtain high permeability in

UHPC and thereby enhance its spalling resistance. Percolation of fiber clusters is also critical to enhancing permeability. The number of fibers that connect to percolating fiber cluster dominates the efficiency of PP fibers on spalling prevention.

Thereafter, a model considering percolation of fiber/crack system is proposed based on the Kozeny-Carman equation to predict permeability of UHPC with fibers. A 3D continuum percolation model is developed to evaluate the percolation and connectivity of fiber/crack system. Finally, a semi-empirical equation is proposed to determine the geometry and dosage of PP fibers for preventing spalling of UHPC.

In the last part, the usage of natural fibers as an alternative to synthetic fibers like PP fibers for preventing spalling of UHPC is explored. The effects of natural fibers on mechanical properties, hot permeability as well as spalling resistance of UHPC are investigated. Furthermore, the effects of accelerated weathering conditions on the performance of natural fibers in UHPC are also evaluated. The findings show that shrinkage of jute fibers at high temperatures enhances the permeability of UHPC and thereby, improves spalling resistance. Accelerated weathering curing affects mechanical properties of UHPC with jute fibers slightly but does not affect spalling resistance of UHPC.

LIST OF PUBLICATIONS

Journal papers:

D. Zhang, A. Dasari, K.H. Tan, On the mechanism of prevention of explosive spalling in ultra-high performance concrete with polymer fibers, *Cement and Concrete Research*, 113 (2018) 169-177.

Conference paper:

D. Zhang, K.H. Tan, A. Dasari, Role of polymer fibres in prevention of explosive spalling in ultra-high performance concrete, in: Ali Nadjai, Faris Ali, Jean-Marc Franssen, O. Vassart (Eds.) *10th International Conference on Structures in Fire SiF'18* Belfast, United Kingdom, 2018, pp. 85-91.

LIST OF TABLES

Table 2.1. Physicochemical change of concrete at elevated temperature (Grattan-Bellew 1996, Zhou and Glasser 2001, Handoo et al. 2002, Alarcon-Ruiz et al. 2005, Mehta and Monteiro 2006, Fares et al. 2010, Neville 2011).	11
Table 2.2. Phase change for PP fibers (Khoury and Willoughby 2008).	34
Table 2.3. Summary of theories and hypotheses on mechanisms of PP fibers in preventing spalling of concrete.	34
Table 3.1. Mixture compositions of UHPC with and without PP fibers (unit in kg/m ³).	48
Table 3.2. Summary of tests.	53
Table 4.1. Mixture compositions.	70
Table 4.2. Connectivity and percolation of fiber clusters.	82
Table 5.1. Properties of cement paste and PP fibers at 150 °C.	104
Table 5.2. Results of spalling tests.	107
Table 5.3. Percolation threshold of fibers used in this study.	108
Table 6.1. Mix proportions of UHPC with different dosages of jute fibers.	116
Table 6.2. Mechanical properties of UHPC after accelerated weathering curing. .	133
Table A.1. Mixture compositions of UHPC.	157
Table A.2. Melting point and CTE of polymer fibers.	160

LIST OF FIGURES

Fig. 1.1. Spalling of concrete columns after fire tests (Kodur and Phan 2007).....	2
Fig. 1.2. Spalling on tunnel segments of the Great Belt tunnel after fire test (Jansson 2013).	3
Fig. 1.3. Spalling damage in the Channel tunnel (Channel Tunnel Safety Authority 1997).	3
Fig. 1.4. Damage and repair work in the Channel tunnel after fire on 11 th September, 2008 (photos by AFP) (Lottman 2017).	3
Fig. 2.1. Residual compressive strength of UHPC at elevated temperature: (a) absolute values and (b) relative values (Poon et al. 2001, Tai et al. 2011, Zheng et al. 2012, Hager et al. 2013, Lai et al. 2013, So et al. 2015).....	12
Fig. 2.2. Tensile strength of UHPC as a function of temperature (Abid et al. 2017).	13
Fig. 2.3. Schematic diagram of thermal stress spalling (Ozawa et al. 2012, Klingsch 2014).	16
Fig. 2.4. Temperature in different location of cylinder specimen (Klingsch et al. 2013).	17
Fig. 2.5. Mechanism of pore pressure spalling (Consolazio et al. 1998).....	19
Fig. 2.6. Moisture clog observed from a spalled slab (Jansson 2013).	20
Fig. 2.7. Test setup for pore pressure measurement (Kalifa et al. 2001).	20
Fig. 2.8. (a) Test setup by Felicetti et al. (2017) (b) specimen with insulation.....	22
Fig. 2.9. Combined pore pressure and thermal stress spalling mechanism (Khoury 2008).	23
Fig. 2.10. Channel created by PP fiber at elevated temperature (Zheng et al. 2014).	36
Fig. 2.11. Thermal analysis of PP fibers (Ozawa and Morimoto 2014).	36

Fig. 2.12. SEM images of PP fiber in concrete: (a) Fiber was visible before heating and (b) fiber disappeared after heated to 200 °C (Kalifa et al. 2001).....	37
Fig. 2.13. Schematic of water droplet test by Kalifa et al. (2001).....	37
Fig. 2.14. Water droplet test on the concrete after being heated (Jansson 2013).	38
Fig. 2.15. SEM micrograph on concrete with PP fibers (a) before and after exposure to 200 °C for 48 hours (Bošnjak et al. 2013).....	39
Fig. 2.16. SEM micrograph by Jansson (2013) showing molten PP flowing out of fiber bed.....	40
Fig. 2.17. Sketch on interconnecting network of microcracks generated in heated concrete (Smith and Atkinson 2010).	41
Fig. 2.18. Development of microcracks in heated concrete by X-ray computed tomography (Pistol et al. 2011).	41
Fig. 2.19. ITZ around PP fibers (Mazzucco et al. 2015).	42
Fig. 2.20. ITZ connection due to PP fibers (Mazzucco et al. 2015).....	43
Fig. 2.21. Formation of PITS (Khoury 2008).....	43
Fig. 3.1. Set-up of TMA.	49
Fig. 3.2. Heating curve of spalling test.....	53
Fig. 3.3. Results of spalling test for (a) UHPC without fibers, and (b) UHPC with fibers subjected to standard ISO 834 heating curve for 1 hour.	55
Fig. 3.4. Residual and hot gas permeability of UHPC with and without fibers and with increasing temperature.....	56
Fig. 3.5. TGA-DSC data of PP fibers used in the current study.	56
Fig. 3.6. XRD spectra of (a) UHPC/O and (b) UHPC/F subjected to different temperatures.....	57
Fig. 3.7. TGA, DTG and DSC results of UHPC/O and UHPC/F.	58
Fig. 3.8. Dimensional (strain) changes of PP, cement paste and aggregate with increasing temperature.....	59

Fig. 3.9. Microstructural evolution in UHPC/F sample with temperature: (a) ambient temperature, (b) 105 °C, (c) 150 °C, (d) 170 °C, (e) 200 °C, and (f) 300°C.	62
Fig. 3.10. Changes in (a) number density, (b) inter-connectivity, and (c) area fraction of microcracks with temperature. The dotted line in (c) considered the areas left by melted fibers.....	64
Fig. 3.11. Microstructural evolution in UHPC/O sample with temperature: (a) ambient temperature, (b) 105 °C, (c) 150 °C, (d) 200 °C, and (e) 300 °C.....	65
Fig. 4.1. X-ray CT system.....	72
Fig. 4.2. Image acquisition of CT images.	73
Fig. 4.3. Connectivity in different UHPC samples.	75
Fig. 4.4. Spalling test results: (a) UHPC-C, (b) U33/12/03, (c) U33/06/03, (d) U100/06/03, (e) U100/12/03, (f) U100/12/06, (g) U100/12/09, and (h) U100/12/12.	77
Fig. 4.5. Permeability of UHPC samples.....	78
Fig. 4.6. Fiber and air bubble distribution in U33/12/03 sample: (a) overview and (b) detail.....	79
Fig. 4.7. Fiber distribution in different UHPC samples: (a) U33/12/03, (b) U33/06/03, (b) U100/06/03, (d) U100/12/03, (e) U100/12/06, (f) U100/12/09, and (g) U100/12/12.	80
Fig. 4.8. Typical connection of two microcracks.....	81
Fig. 4.9. Cumulative percentage as a function of fiber distance in UHPC (a) with different fiber geometry and (b) different fiber dosages.	83
Fig. 4.10. Distance distribution of fiber clusters of UHPC with (a) different fiber geometry and (b) fiber dosage.	84
Fig. 4.11. Percolation of fiber cluster in UHPC samples: (a) U33/12/03, (b) U100/12/09, and (c) U100/12/12. Percolating fiber cluster is in green and isolated fiber cluster is in red.	86

Fig. 4.12. Air bubbles in U33/12/03 sample.....	87
Fig. 4.13. Air bubble content of UHPC samples.	88
Fig. 4.14. Flow of UHPC samples.....	89
Fig. 4.15. Compressive strength of UHPC with different sizes and different dosages of fibers.....	89
Fig. 5.1. Sketch of microcrack in concrete: (a) cross-section and (b) microcrack along fiber.....	95
Fig. 5.2. SEM micrograph on microcrack along fiber channel (After exposure to 150 °C, UHPC samples were cut and polished. The fiber remained in the channel was removed).....	96
Fig. 5.3. Sketch of percolation model.....	100
Fig. 5.4. An example of results of 100 implementations (element size: $l_f = 12$ mm, $h_c = 185$ μ m, total number of fibers = 1.313×10^4).....	101
Fig. 5.5. Histogram of fraction of percolating fibers for 100-time calculation (element size: $l_f = 12$ mm, $h_c = 185$ μ m, total number of fibers = 1.313×10^4).	102
Fig. 5.6. The setup of three-point bending test and the specimen geometry.	104
Fig. 5.7. Permeability with increased temperature: (a) UHPC with 28 μ m PP fibers, (b)UHPC with 33 μ m PP fibers and (c)UHPC with 100 μ m PP fibers.	106
Fig. 5.8. Percolation threshold of fibers with different aspect ratios (l_f / d_f).	108
Fig. 5.9. Comparison of predicted results and test results.	109
Fig. 5.10. Relationship between C_{PBN} and l_f / d_f	110
Fig. 5.11. Predicted permeability with different fiber geometries.....	111
Fig. 5.12. Comparison of the spalling test results and C_{PBN} curve (green symbol: no spalling, red symbol: spalling).	112
Fig. 6.1. Jute fibers used in this study.....	116
Fig. 6.2. SEM micrographs of the jute fiber: (a) surface and (b) cross-section.	116
Fig. 6.3. Mass loss after exposure to different temperatures.	121

Fig. 6.4. Failure pattern of (a) UHPC-C and (b) UHPC-J10 after compressive strength.	121
Fig. 6.5. Residual compressive strength of UHPC with jute fibers exposed to high temperature.	123
Fig. 6.6. TGA-DSC results of (a) jute fibers and (b) UHPC with different dosages of jute fiber.	124
Fig. 6.7. Residual flexural strength of UHPC with jute fibers exposed to high temperature.	126
Fig. 6.8. TMA result of jute fiber and cement matrix.	128
Fig. 6.9. SEM micrograph of jute fiber in UHPC at (a) ambient temperature and (b) 200 °C.	128
Fig. 6.10. Spalling test results of UHPC with different jute fiber dosages: (a) UHPC-C, (b) UHPC-J3, (c) UHPC-J5, and (d) UHPC-J10.	129
Fig. 6.11. Hot permeability of UHPC with different fiber dosages.	130
Fig. 6.12. Percolation of jute fibers in (a) UHPC-J5 and (b) UHPC-J10 (the volume of prism: 7 mm × 7 mm × 15.7 mm).	130
Fig. 6.13. XRD results of UHPC-J10 samples after accelerating aging.	132
Fig. 6.14. SEM micrograph of jute fiber in UHPC after accelerated weathering curing: (a) UHPC-J10-SD and (b) UHPC-J10-QUV.	132
Fig. 6.15. Spalling test after accelerated weathering curing: (a) UHPC-J10-SD and (b) UHPC-J10-QUV.	133
Fig. A.1. Spalling test results: (a) UHPC-LLDPE, (b) UHPC-UHMWPE, (c) UHPC-PA66 and (d) UHPC-PET.	158
Fig. A.2. DSC results of different polymer fibers.	159
Fig. A.3. Thermal strain of polymer fibers with increasing temperature.	159
Fig. A.4. SEM images of UHPC with polymer fibers: (a) UHPC-LLDPE after exposure to 105 °C, (b) UHPC-UHMWPE after exposure to 105 °C, (c) UHPC-PA66	

after exposure to 200 °C, and (d) UHPC-PET after exposure to 200 °C.....	160
Fig. A.5. Hot permeability of UHPC with different polymer fibers.....	162
Fig. A.6. SEM micrographs of (a) UHPC-LLDPE and (b) UHPE-UHMWPE after exposure to 200 °C.....	162

LIST OF ABBREVIATIONS

The following list of abbreviations is provided for ease of reference for those abbreviations that are most frequently used in this thesis. It is therefore not exhaustive. Nonetheless, all symbols and abbreviations used in this thesis are defined at their first mention in the main text.

3D: three dimensional

CH: calcium hydroxide.

C₂S: dicalcium silicate

C₃S: tricalcium silicate

C-S-H: calcium silicate hydrate

CTE: coefficient of thermal expansion

DMA: dynamic mechanical analyzer

DSC: differential scanning calorimeter

HSC: high strength concrete

ITZ: interfacial transition zones

LLDPE: linear low-density polyethylene

NSC: normal strength concrete

PA: polyamide

PE: polyethylene

PET: polyester

PITS: pressure-induced tangential space

PP: polypropylene

PVA: Polyvinyl alcohol

SCC: self-compacting concrete

SEM: scanning electron microscopy

TGA: thermogravimetric analysis

TMA: thermomechanical analyzer

UHMWPE: ultra-high molecular weight polyethylene

UHPC: ultra-high performance concrete

W/B ratio: water to binder ratio

W/C ratio: water to cement ratio

X-ray CT: X-ray computed tomography

XRD: X-ray diffraction

CHAPTER 1 INTRODUCTION

1.1 Research background

Concrete is an essential material for construction and building industries. Recently, great effort has been devoted to the development of high strength concrete. Within the last three decades, ultra-high performance concrete (UHPC) with a compressive strength of more than 150 MPa and high durability has been developed. With high strength, UHPC can offer significant economic, architectural, and structural advantages over conventional concrete. For example, the superior strength of UHPC helps reduce component sections significantly, leading to a reduction in weight and an increase in usable floor space, especially in high-rise buildings (Tam et al. 2012). In addition, enhancing concrete strength is also considered as an important strategy to reduce environmental footprint during the production of cement, which may contribute to 5-10 % of the global anthropogenic carbon dioxide production (Qomi et al. 2014). Furthermore, the high density and low porosity of UHPC make it nearly impermeable to carbon dioxide, chlorides, and sulfates. As UHPC has high abrasion resistance and corrosion resistance, it will enhance the long-term durability of structures that are exposed to corrosive environments.

UHPC has been used in many engineering projects such as bridges and nuclear waste storage structures in the last two decades (Shi et al. 2015). However, due to its dense microstructure, UHPC is highly susceptible to explosive spalling under fire condition. As shown in Fig. 1.1, explosive spalling leads to a sudden loss of concrete, reduces the cross-sections of members and exposes inner layers of reinforcement to high temperature, hence compromising the member load capacity (Kodur and Phan 2007). Damages caused by spalling may significantly affect the fire resistance of concrete

structure.

Fig. 1.1. Spalling of concrete columns after fire tests (Kodur and Phan 2007).

In 1994, a fire occurred in Great Belt Tunnel and lasted for 4 to 8 hours. The concrete segment spalled severely. The concrete segment spalled severely and only one-third of the lining section remained. After the fire incident, two tunnel elements with the same mix design as those in Great Belt Tunnel were produced and used in the fire tests conducted at SP Technical Research Institute of Sweden. The tunnel element from the Great Belt tunnel showed severely spalling after testing, as shown in Fig. 1.2.

During the fire in the Channel Tunnel on 18th November, 1996, spalling of concrete also caused severe damage to the lining segment, as shown in Fig. 1.3. Extensive damage was observed along 50 m of the tunnel. At some places, 450 mm thick concrete elements almost spalled completely (Channel Tunnel Safety Authority 1997, Khoury and Anderberg 2000). Another fire in the Channel tunnel on 11th September, 2008, also caused severe spalling of the tunnel lining, as shown in Fig. 1.4. The

spalling occurred along 284 m of the tunnel (BEA-TT & RAIB 2010).

Fig. 1.2. Spalling on tunnel segments of the Great Belt tunnel after fire test (Jansson 2013).

Fig. 1.3. Spalling damage in the Channel tunnel (Channel Tunnel Safety Authority 1997).

Fig. 1.4. Damage and repair work in the Channel tunnel after fire on 11th September, 2008 (photos by AFP) (Lottman 2017).

It is believed that thermal stress (generated by temperature gradients) and high vapor pressure are two main mechanisms of spalling (Khoury 2000, Ozawa and Morimoto 2014, Ma et al. 2015). To prevent spalling, one of the most widely accepted methods is the inclusion of polymer fibers. It is commonly agreed that adding polypropylene (PP) fibers could increase permeability of concrete and hence reduce spalling. (Kalifa et al. 2001, Lee et al. 2012). Other fibers, such as polyethylene and steel fibers, are also used in concrete for spalling prevention. Additionally, most of the previous studies are based on normal strength concrete (NSC) or high strength concrete (HSC). Whether these conclusions can be adopted for spalling prevention of UHPC needs to be investigated. Furthermore, findings of experimental studies from different researchers are not consistent and at times even contradictory. This is mainly due to a lack of thorough understanding of the mechanism of polymer fibers in preventing spalling. Choosing fibers for preventing spalling still mainly relies on trial-and-error approach. In addition, other factors, such as various mix design and different experimental methods, also affect test results. Up till now, it is still challenging to optimize fiber geometry and dosage for spalling prevention of UHPC.

Consequently, further experimental investigations are required to gain deeper insight into the mechanism of polymer fibers on preventing spalling. The mechanism of fiber geometry affecting spalling resistance needs to be investigated in detail. This knowledge is of great scientific interest and forms the basis for a targeted fiber optimization (both geometry and dosage) and for the search for cheaper alternatives. Additionally, important parameters can be identified for developing a targeted specific fiber or other materials.

1.2 Scope, methodology, and novelty of research

The scope of work can be classified as shown below:

- (1) To gain an overall understanding of the behavior of UHPC at high temperature, particularly, explosive spalling.
- (2) To thoroughly investigate and understand the exact role of polymer fibers in the prevention of explosive spalling in UHPC.
- (3) To investigate the effect of fiber geometry and fiber dosage on the spatial distribution of fibers in concrete, and its effect on spalling resistance of UHPC.
- (4) To develop a method for determining the critical fiber geometry and dosage for spalling prevention of UHPC.
- (5) To evaluate the potential of using other fibers, especially plant-based natural fibers that are environmentally friendly, to prevent spalling of UHPC.

A range of analytical and microscopic tools are used to realize the objectives of research. Thermal gravimetric analyzer (TGA), differential scanning calorimetry (DSC), X-ray diffraction (XRD) and thermomechanical analysis (TMA) are adopted to analyze phase/chemical change of UHPC and fibers. Scanning electron microscopy (SEM) is used to characterize the microstructural changes in UHPC before and after exposure to high temperature. The impact of microcracks evolution on permeability of UHPC under high temperature is unveiled through quantitative investigation on the crack geometry. Fiber distribution is characterized in 3D by X-ray computed tomography (CT). The connectivity and distribution of fibers in concrete samples are quantitatively analyzed. Mechanical properties, such as compressive strength, flowability, flexural strength, and permeability, are also measured to study the effect of fiber inclusion on the properties of UHPC. Spalling tests in this work are conducted following the ISO834 fire curve. A model based on the Kozeny-Carman equation is developed to predict the permeability of UHPC. A

3D percolation model is also proposed to calculate the percolation threshold and connectivity of fiber/crack system.

The findings of this research work are novel as they clarify the exact role of polymer fibers in spalling prevention of UHPC. For the first time, it characterizes the spatial distribution of polymer fibers in UHPC and correlates it to spalling resistance. Percolation threshold and connectivity of fiber/crack system are considered as key factors for preventing spalling. In addition, a simple method considering fiber geometry and fiber dosage is proposed to produce non-spalling UHPC. Furthermore, plant-based natural fiber is used as a new alternative, which is economical and environmentally friendly.

1.3 Layout of the report

This thesis consists of seven chapters. This first chapter gives a general introduction of the research background and the motivation of this study. Thereafter, the objectives, scope of work, methodology, and novelty are presented.

In Chapter 2, an extensive literature review on UHPC at high temperature is carried out. Mechanical properties, physicochemical and microstructural changes of concrete exposed to high temperature are first introduced. Then, a critical overview on explosive spalling is presented, including some important fire tests and mechanisms of spalling. Afterwards, various proposed hypotheses on mechanisms of PP fibers in preventing spalling are summarized. Finally, previous studies and optimization on different types of fiber for spalling prevention are discussed.

In Chapter 3, an in-depth re-examination on the mechanism of polymer fibers in spalling prevention of UHPC is presented. A series of experiments on UHPC with and without PP fibers are conducted. Spalling and permeability tests are performed,

followed by investigations of microstructural changes in UHPC samples with and without fibers. A stereological method is employed to quantify microcrack geometry in UHPC and clarify the effect of PP fibers on preventing spalling.

In Chapter 4, an investigation on the spatial distribution of PP fibers in UHPC and its effect on spalling resistance is carried out. Different fiber geometry and fiber dosages are used in UHPC mixes in this work. The influences of fiber geometry and dosage on the connectivity and distribution of fibers in UHPC are discussed.

In Chapter 5, the importance of percolation and connectivity of fiber/crack system in preventing spalling is further discussed. A 3D continuum percolation model is developed to evaluate percolation and connectivity of fiber/crack system in UHPC. Then, an approach to predict permeability of UHPC with PP fibers is proposed based on the Kozeny-Carman equation. Finally, a semi-empirical equation is proposed to determine the geometry and dosage of PP fiber for preventing spalling of UHPC. The approach proposed in this chapter is validated by permeability and spalling test results.

In Chapter 6, the effects of natural fibers on mechanical properties, hot permeability along with spalling resistance of UHPC are investigated. Additionally, preliminary short-term durability tests were also conducted to study the changes in properties of UHPC with fibers.

In Chapter 7, the conclusions of current research work are summarized and the plan for future work is proposed.

CHAPTER 2 LITERATURE REVIEW

2.1 Introduction

In this chapter, a review of previous studies related to UHPC at high temperature is presented. This constitutes the development of UHPC, thermal degradation of UHPC and NSC, and their spalling responses. The different proposed mechanisms for explosive spalling of concrete are also discussed. Thereafter, attention is given to methodologies that are employed to prevent spalling of concrete. Emphasis has been put on the mechanism of spalling prevention by incorporation of polymer fibers.

2.2 Development of UHPC

de Larrard and Sedran (1994) first introduced the term “ultra-high performance concrete”, which defines concrete with high compressive strength, high toughness and excellent durability (Shi et al. 2015). Generally, UHPC represents the type of concrete with a cylinder compressive strength of more than 150 MPa. In some publications, concrete with a compressive strength exceeding 120 MPa is also considered as UHPC (Kodur 2014).

High content of binder and low water-to-binder ratio are required to produce UHPC. Generally, the binder content in UHPC is around 1000 kg/m³ and the water-to-binder ratio is usually below 0.2 (Shi et al. 2015). The absence of coarse aggregates and the presence of proper amounts of supplementary cementitious materials like fly ash, silica fume, and granulated blast furnace slag (GGBS) is believed to improve the homogeneity, and thereby high strength of UHPC (in the range of 150 MPa to 200 MPa) (Shi et al. 2015, Wang et al. 2015). Additionally, low porosity and dense microstructure are other factors leading to the high strength of UHPC (Wu et al. 2018).

Furthermore, higher compressive strength, ranging from 200 MPa to 650 MPa, was also reported by using thermal curing at 90 °C or higher, vacuum mixing, and applying pressure before and during setting of concrete (Richard and Cheyrezy 1995, Shi et al. 2015, Wang et al. 2015).

2.3 Physiochemical change, microstructure and mechanical properties of UHPC at high temperature

High temperature causes phase transformations and chemical reactions in concrete, resulting in progressive changes of the microstructure of UHPC (Handoo et al. 2002). A list of the proposed changes with temperature is given in Table 2.1. These changes affect properties like compressive strength and splitting tensile strength (Abid et al. 2017). For example, in Fig. 2.1, a collection of data from different studies is plotted, highlighting how the compressive strength of UHPC varies with temperature. Generally, three stages can be identified:

- (1) From ambient temperature to ~400 °C: Compressive strength slightly increases or remains constant with an increase of temperature. The exact reason for the change is unclear. In some studies, this gain is attributed to dry hardening, phase changes induced by hydrothermal conditions, the “internal autoclave” effect of vapor, and additional hydration of cement particles (Dias et al. 1990, Phan 1996, Behnood and Ziari 2008, Rashad et al. 2012, Rashad and Zeedan 2012, Zheng et al. 2014, Sanchayan and Foster 2015, Abid et al. 2017).
- (2) From 400 °C to 800 °C: A significant reduction in compressive strength occurs above 400 °C. By 600 °C, a reduction of ~50 % of initial strength is generally noted. This loss in compressive strength is attributed to the decomposition of calcium-silicate-hydrate (C-S-H) and calcium hydroxide (CH) (Zheng et al. 2013,

Zheng et al. 2014). It was also reported that the strength reduction could be caused by the phase transformation of quartz from α to β form, which results in an expansion of volume (0.8 %), and hence creates cracks in concrete (Fares et al. 2010).

- (3) From 800 °C onward: The decreasing rate of strength slows down, but UHPC loses most of the compressive strength, leaving only around 20 % of the original value.

Table 2.1. Physicochemical change of concrete at elevated temperature (Grattan-Bellew 1996, Zhou and Glasser 2001, Handoo et al. 2002, Alarcon-Ruiz et al. 2005, Mehta and Monteiro 2006, Fares et al. 2010, Neville 2011).

Temperature (°C)	Reaction and characteristic
30–105	<ul style="list-style-type: none"> • Escape of free water and a part of physically bound water
110–170	<ul style="list-style-type: none"> • Decomposition of gypsum (with a double endothermic reaction) • Decomposition of ettringite • Loss of water from parts of the carbo-aluminate hydrates
180–300	<ul style="list-style-type: none"> • Loss of chemically bound water from the decomposition of C-S-H and carbo-aluminate hydrates
450–600	<ul style="list-style-type: none"> • Dehydroxylation of CH ($\text{Ca(OH)}_2 \rightarrow \text{CaO} + \text{H}_2\text{O}$) • Transformation of quartz in aggregates from α to β form with volumetric expansion
600–900	<ul style="list-style-type: none"> • Decarbonization of calcium carbonate • Decomposition of C-S-H

Fig. 2.1. Residual compressive strength of UHPC at elevated temperature: (a) absolute values and (b) relative values (Poon et al. 2001, Tai et al. 2011, Zheng et al. 2012, Hager et al. 2013, Lai et al. 2013, So et al. 2015).

Unlike compressive strength, most studies showed that the tensile strength of UHPC decreases linearly with increasing temperature (Fig. 2.2) (Tai et al. 2011, So et al. 2015, Xiong and Liew 2015, Abid et al. 2017).

Fig. 2.2. Tensile strength of UHPC as a function of temperature (Abid et al. 2017).

2.4 Spalling of concrete

2.4.1 Definition and types of spalling

According to Khoury (2008), spalling of concrete is defined as “the violent or non-violent breaking off of layers or pieces of concrete from the surface of a structural element, when it is exposed to high and rapidly rising temperature as experienced in fires”.

From the experimental observation, spalling of concrete at high temperature is usually categorized into six types: (1) explosive spalling, (2) surface spalling, (3) aggregate spalling, (4) corner spalling, (5) post-cooling spalling, and (6) sloughing-off spalling (Meyer-Ottens 1972, Khoury and Anderberg 2000, Klingsch 2014). This categorizing method covers all the observations from the tests. It is widely accepted by researchers.

Generally, the violent and non-violent spalling in the definition is based on the energy

released during spalling. But there is no quantitative criterion that can be used to distinguish them. Sloughing-off and post-cooling spalling usually occur with concrete parts falling off from the structure members. They usually start after a long duration of exposure or even after the temperature of concrete cools to ambient temperature. The energy released in these two types of spalling is relatively low. Therefore, they are usually considered as non-violent spalling. In fact, these two types of spalling rarely occur. On the other hand, surface and explosive spalling are considered to be violent. Explosive, surface, and aggregate spalling usually occur at the early stage of fire exposure (within 30 min), accompanied by a sudden release of high internal stresses. Corner spalling may occur after 30-90 min of fire exposure. In reality, several different types of spalling may occur simultaneously under fire condition, and it is difficult to distinguish them. In literature, surface, corner, and explosive spalling were simply unified as explosive spalling (Klingsch 2014).

Among these types of spalling, explosive spalling is the most serious. When HSC and UHPC members suffer explosive spalling under fire condition, it can cause rapid removal of concrete layers, leading to high heat flow to the inner concrete or reinforcing members (Kodur and Phan 2007). Both the reduction of members' cross-section and the sharp increase of temperature on reinforcement bars affect the load-carrying capacity of structures significantly (Kodur 2000). Considering this, the research work presented in this thesis will mainly focus on explosive spalling of UHPC.

2.4.2 Mechanisms of explosive spalling

2.4.2.1 Overview

There are many studies on the elaboration of the mechanisms of spalling. However,

the mechanisms are not fully understood due to the complexity of the whole process. Some hypotheses, including thermal stress theory, pore pressure theory, and a combination of both theories are discussed in this section to provide a comprehensive understanding of concrete spalling under fire.

2.4.2.2 Thermal stress theory

In this theory, thermal stresses caused by rapid heating is considered as the main reason for spalling (Khoury and Anderberg 2000). When concrete is exposed to high temperature, rapid heating generates a thermal gradient in the cross-section of concrete, which then induces compressive stresses near the heated surface and tensile stress in the cooler interior regions (Ozawa et al. 2012). The compressive stress lies parallel to the heated surface while the tensile stress is in the perpendicular direction, as illustrated in Fig. 2.3. Therefore, according to Saito (1966), thermal-stress-induced spalling was due to compression failure of concrete at the heated surface. Applied load, heating rate, compressive strength, and tensile strength of concrete play an important role in thermal-stress-induced spalling (Khoury 2008).

This theory has been discussed by many researchers. Some studies on testing different sizes of specimens provided evidence of thermal stress spalling. Guerrieri et al. (2008) prepared a series of concrete slabs of 780 mm × 360 mm with different thicknesses, which were 100, 200, and 400 mm. The specimens, with a compressive strength of 75 MPa, were exposed to hydrocarbon fire on one side. Only the slab with a thickness of 400 mm suffered spalling during fire exposure. They suggested that thermal stress played a critical role in explosive spalling. Heo et al. (2014) also held a similar view through testing concrete square columns with different cross-sections, viz. 300, 500, 700, and 900 mm. The results indicated that the larger the size of a column, the greater the severity of spalling was. They considered that this was caused by the higher

temperature gradient in a larger column section. Another study conducted by Han et al. (2009) found that when a thermal barrier was used to protect concrete, the falling off and failure of the thermal barrier could cause a sudden increase in the temperature of the concrete surface, which might lead to severe spalling. This test also indicated the effect of thermal gradient on the spalling of concrete. In some numerical studies on explosive spalling of HPC specimens, it was also shown that thermal stress caused by temperature gradient is the main reason of spalling (Zhang and Davie 2013, Zhao et al. 2014).

Fig. 2.3. Schematic diagram of thermal stress spalling (Ozawa et al. 2012, Klingsch 2014).

It should be noted that thermal stress theory cannot explain some spalling behavior that occurred at a very low heating rate (Jumppanen 1989, Hertz 1992, Hertz 2003, Fu and Li 2011, Klingsch et al. 2013, Klingsch 2014). For example, Klingsch et al.

(2013) observed spalling even with a very low heating rate of 1 °C/min. At such a low heating rate, thermal gradient was negligible, as shown in Fig. 2.4, and thermal stress caused by temperature gradient was insignificant. Similarly, in the tests conducted by Hertz (1992), UHPC samples also spalled at a heating rate of 1 °C/min. In fact, Klingsch (2014) questioned that whether thermal stresses generated by temperature gradient near the heated surface could be high enough to cause compressive failure, especially in UHPC samples with high compressive strength of more than 150 MPa. However, none of the studies have validated this theory through experimental measurements.

Many investigations also found that water-to-cement (W/C) ratio, moisture content and silica fume content may affect spalling resistance of UHPC (Shorter and Harmathy 1961, Zhukov 1976, Hertz 1984, Klingsch et al. 2013).

Fig. 2.4. Temperature in different location of cylinder specimen (Klingsch et al. 2013).

2.4.2.3 Pore pressure theory

Shorter and Harmathy (1961) found that only concrete of a certain humidity suffered from spalling. It was found that if the specimens were dried to a depth of 50 to 65 mm from the surface, spalling could be avoided. Dry concrete specimens did not spall even under a large temperature gradient. They considered that moisture was the more critical factor for inducing spalling and thermal stress might not induce spalling of concrete in the absence of moisture. This forms the basis of pore-pressure induced spalling (Hertz 2003).

Pore pressure spalling results from the build-up of high pore pressure, associated with migration of mass, including air, vapor and liquid water in the porous network of concrete (Consolazio et al. 1998). The process of build-up pressure and pore pressure spalling is shown in Fig. 2.5. Water is evaporated during the heating period, generating significant pressure in concrete. Driven by the pressure gradient, migration of vapor occurs in the zone near the heated surface. Not only does the vapor transport outwards, but it also migrates inwards into the cooler interior concrete where it condenses due to lower temperature. As vapor migration and condensation continue, water accumulates at the cooler region and finally forms a saturated layer, which is known as a “moisture clog” (Harmathy 1965, Harmathy 1966), as shown in Fig. 2.5. The saturated layer stops the vapor from further migrating into the cooler interior region. As a result, a rapid rise in pore pressure is generated right in front of the saturated layer due to the accumulation of vapor. Spalling may occur when this pore pressure exceeds the tensile strength of concrete (Kalifa et al. 2000, Fu and Li 2011, Ozawa et al. 2012).

Fig. 2.5. Mechanism of pore pressure spalling (Consolazio et al. 1998).

Jansson (2013) observed the moisture clog by splitting a spalled slab, as shown in Fig. 2.6. Chen et al. (2009) also found the gas retention phenomenon by measuring continuous gas permeability during heating, providing experimental evidence for the moisture clog theory. In an investigation by der Heijden et al. (2012), evidence of moisture clog was obtained by nuclear magnetic resonance spectroscopy (NMR). In recent years, some studies have been conducted to measure the pore pressure and temperature at different depths from the heated surface in concrete specimens (Kalifa et al. 2000, Kalifa et al. 2001, Mindeguia et al. 2010, Bangi and Horiguchi 2011, Bangi and Horiguchi 2012, Ding et al. 2016). An example of the experimental set-up (by Kalifa et al. (2000)) is shown in Fig. 2.7. The test results clearly showed that the pore pressure peak and gradient were much higher in HSC than those in NSC, and chemically-bound water in cement matrix was sufficient to form the moisture clog, as spalling also occurred in samples with low initial water content (Kalifa et al. 2000).

Fig. 2.6. Moisture clog observed from a spalled slab (Jansson 2013).

Fig. 2.7. Test setup for pore pressure measurement (Kalifa et al. 2001).

In the first fire tests on large concrete structures regarding spalling conducted by Gary (1918), it was reported that concrete with high water content was more susceptible to spalling. Furthermore, Zhukov (1976) found that W/C ratio could be a critical factor that affects the spalling risk of concrete. High W/C ratio led to high porosity and permeability, hence reducing the risk of spalling. By contrast, a low W/C ratio resulted in high spalling risk. A similar observation has been reported by Malhotra (1984), who found that dense concrete with low permeability showed high vapor pressure during heating and high risk of spalling. Based on Malhotra's work, permeability of concrete was considered as an important parameter in spalling of concrete. In addition, Hertz (1992) found that adding silica fume in the mix could densify concrete and increase the risk of spalling. Hertz concluded that increasing

moisture content, decreasing permeability, and decreasing tensile strength may increase the risk of explosive spalling. He also found that chemically bound water was sufficient to induce explosive spalling when the concrete microstructure was dense enough (Hertz 1984, Hertz 2003, Hertz 2005). In a recent work, Persson (2004) conducted a series of fire tests on SCC pre-stressed columns and suggested that high values of cement/powder ratio and W/C ratio were required to produce a concrete mix design with good spalling resistance. All of these studies were related to pore pressure induced spalling. Microstructure of concrete and moisture content could affect the accumulation of vapor pressure. Generally, pore pressure induced spalling is influenced by factors including permeability of concrete, moisture content, and tensile strength of concrete (Khoury 2008). Among them, permeability is considered as the main factor. The lower the permeability of a heated specimen is, the sooner the moisture clog forms and the greater the induced pore pressure is (Kalifa et al. 2000, Kalifa et al. 2001). This theory also explains why UHPC and HSC are susceptible to a higher risk of spalling than NSC.

However, Felicetti et al. (2017) devised a special test setup to investigate the effect of pore pressure on fracture behavior of concrete at high temperature. In the test, a cubic specimen was sealed and covered by thermal insulating materials and then placed between two heating panels (as shown in Fig. 2.8). A pressure probe and a thermocouple were used to measure the pore pressure and temperature at the center of the cube during heating. When the pore pressure reached the peak value, the insulations were removed and splitting tests were conducted on the specimens at hot state. The specimens were heated under different heating rates, including 1, 2, 10 and 120 °C/min, to get different values of peak pore pressure. During the tests, they found that the specimens did not spall at high temperature. But when an external load was applied, the hot specimens split suddenly with a loud sound. It was concluded that pore pressure itself could not trigger spalling at high temperature. It should be

combined with thermal stress or external load to trigger spalling.

Fig. 2.8. (a) Test setup by Felicetti et al. (2017) (b) specimen with insulation.

2.4.3 Combined pore pressure and thermal stress spalling

Some studies have pointed out that explosive spalling generally occurs under the combined effect of pore pressure and thermal stress (Khoury and Anderberg 2000). Fig. 2.9 illustrates the mechanism of combined pore pressure and thermal stress spalling presented by Khoury (2000). During heating, cracks develop in the specimen parallel to the heated surface when the tensile stress exceeds the tensile strength of concrete. This is accompanied by a sudden release of energy and violent failure of the heated surface region (Khoury 2008, Lee 2008).

Fig. 2.9. Combined pore pressure and thermal stress spalling mechanism (Khoury 2008).

The finding that spalling was caused by the combined effect of pore pressure and thermal stress was first proposed by Zhukov (1976). It was considered that thermal expansion and applied load could cause uniform compression stress and cracks in the longitudinal direction of heating, while high pore pressure could create additional tensile stress perpendicular to the heated surface and cause spalling.

Sertmehmetoglu (1977) further modified the hypothesis of combined pore pressure and thermal stress spalling based on ideas from Dougill (1971). Dougill noted that cracks could develop parallel to the heated surface due to stress caused by thermal gradient. Sertmehmetoglu speculated that these crack would be filled with vapor. The

vapor pressure in the cracks then created forces normal to the heating face, resulting in spalling. Khoury (2008) presented a general sketch of this mechanism as shown in Fig. 2.9. However, they also mentioned that the governing mechanism could change with different concrete strengths.

In summary, based on the literature review on spalling of concrete and mechanisms, it is found that factors like W/C ratio, content of silica fume, specimen thickness, permeability, tensile/compressive strength, exposed heating rate, and moisture content affect the occurrences of spalling. The governing spalling mechanisms like thermal stresses, pore pressure and combination of both pore pressure and thermal stresses change in concrete with different properties. However, based on the proposed mechanisms, some methods can be considered for spalling prevention: (1) increasing permeability to help release trapped vapor pressure, (2) increasing tensile strength of concrete and (3) reducing heating rate. In Section 2.5, a review on spalling prevention will be presented.

2.5 Spalling prevention

Many preventive measures have been studied and discussed for preventing spalling. According to the literature, utilization of protective lining, thermal barrier, steel fibers, and polymer fibers are recommended for spalling prevention.

2.5.1 Utilization of protective lining and thermal barrier

Protective layer is one of the recommendations proposed by EN 1992-1-2 (British Standards Institution 2005) for spalling prevention. Protective lining usually acts as a thermal barrier to reduce temperature and heating rate on the concrete surface. Klingsch et al. (2013) conducted a series of tests to study the effectiveness of

protective linings on spalling prevention of HPC. Different protective linings and different thicknesses of specimens were discussed. The tests confirmed that protective linings could enhance spalling resistance, but their effectiveness depends on their thickness. Han et al. (2009) also conducted some tests with applying different protective coatings on concrete. They found that spalling was likely to occur at the four edges of the specimens even under the protection of coating. In addition, the adhesion of protective coating at high temperatures was critical to spalling prevention. Delamination coating at high temperature could lead to even more serious spalling.

Besides protective lining, some materials like gypsum boards were also used as thermal barriers for spalling prevention. Han et al. (2009) showed that gypsum boards could reduce the temperature and improve spalling resistance of concrete. However, the assembling method of gypsum boards was important. Spalling might still occur when the gypsum boards cracked or failed.

Generally, the protective linings and thermal barriers were usually used to enhance fire resistance of existing concrete structures. Currently, there are limited design criteria regarding their applications on concrete structures.

2.5.2 Utilization of steel fibers

In many studies, steel fiber was incorporated into concrete for preventing spalling. It is commonly held that steel fiber can increase the tensile strength of concrete to about 5 to 7 MPa, which may be sufficient to achieve two to three hours of fire resistance without significant spalling (Kodur 2000). Some experiments conducted by Kodur and Mcgrath (2003) showed that adding steel fibers improved the fire resistance of concrete members.

In a study by Kahanji et al. (2016), four UHPC beams with 2 % and 4 % steel fibers

were tested under ISO 834 for one hour with three different loading levels of 0.2, 0.4, and 0.6. The compressive strength of concrete was 178 MPa. During heating, all the beams suffered spalling. It was found that increasing steel fiber dosage from 2% to 4 % could reduce spalling up to 87%. It was concluded that the increase in tensile strength of concrete caused by the addition of steel fibers could help UHPC beams withstand the built-up vapor pressure at high temperature. In contrast, some investigations showed that steel fibers had no significant influence on spalling resistance. Hertz (1992) investigated the effect of steel fibers on spalling resistance of concrete with a compressive strength of 170 MPa. Experimental results showed that adding steel fibers only delayed spalling from 300 °C to 400 °C but did not prevent spalling. Even worse, specimens with 3 % (by weight) of steel fibers showed the highest possibility of spalling among the specimens with 0, 1.5 and 3 % (by weight) of steel fibers. Klingsch (2014) tested concrete samples containing 2.5 % (by volume) of steel fibers. Two mixes of concrete (compressive strength: 151.3 MPa and 96.5 MPa) were tested respectively. It was found that steel fibers could not increase spalling resistance of UHPC. Sideris et al. (2009) also stated that explosive spalling still occurred even though adding steel fibers could increase residual tensile strength of concrete up to 300 °C. Furthermore, recent studies by Li et al. (2019) showed that severe spalling occurred in all the UHPC samples with only steel fibers. Spalling resistance of UHPC was not improved even with 236 kg/m³ of steel fibers. Also, there was no enhancement in permeability and reduction of pore pressure of UHPC samples with steel fibers at high temperature (Li et al. 2018, Li et al. 2019).

In short, according to previous works on steel fibers in UHPC, most of the test results showed that adding steel fibers was not an effective method to eliminate spalling of UHPC. Adding steel fibers may reduce the extent of spalling or delay spalling to higher temperatures but cannot prevent spalling.

2.5.3 Utilization of polymer fibers

Polymer fibers such as polypropylene (PP), polyvinyl alcohol (PVA) fibers, polyamide (PA), and polyethylene (PE) fibers, have also been used for preventing spalling of concrete in recent studies (Heo et al. 2010, Missemer et al. 2010, Ozawa and Morimoto 2014, Sanchayan and Foster 2015).

The effectiveness of four different synthetic fibers, including PP, PE, PVA and acrylic (PAN) fibers, on preventing spalling of UHPC was studied (Missemer et al. 2010, Missemer et al. 2010, Missemer et al. 2019). A blowtorch test was conducted to investigate the spalling behavior of concrete with different fiber types. It was found that both PP and PE fibers were effective for spalling protection but PVA fibers did not show the same benefit as PP and PE fibers. It was considered that this was caused by the high melting temperature of PVA fibers (about 240 °C). Moreover, PAN fibers were found to be inefficient, even though they had a lower melting temperature (120 °C). Microscopy observation demonstrated that a film was left in the bed by PAN fibers after they had melted and the cracks in PAN fibers' beds were much less pronounced than those in PP fibers' beds.

PE fibers are widely used to enhance mechanical properties of concrete. Due to their high strength and high modulus, PE fibers could be used to develop high-strength high-ductility cementitious composites (Ranade et al. 2013, Ranade et al. 2013, He et al. 2017). Recent studies found that although PE fiber had a melting point of 145 °C, which was even lower than that of PP fiber (melting point: round 170 °C), it did not improve spalling resistance of UHPC even with a high fiber content (Liu et al. 2018). In a test conducted by Li (2018), spalling of UHPC was observed in the samples with 1 vol.% of PE fibers.

Although many polymer fibers were studied for preventing spalling in UHPC, the findings are still limited. Obviously, the test results showed that melting point of fiber cannot determine the effectiveness of fibers in spalling prevention. However, till now, the researchers could not identify the main attributes of polymer fibers responsible for spalling prevention.

Among various types of polymer fibers, PP fiber is one of the most widely used fibers. Many studies have shown that adding PP fibers was effective in preventing spalling of NSC and HSC (Diederichs et al. 1993, Kalifa et al. 2001, Persson 2004, Boström and Jansson 2008). It is also recommended in EN 1992-1-2 (British Standards Institution 2005) that at least 2 kg/m^3 (or equivalent 0.2 vol.%) of monofilament PP fibers should be included in the concrete mix. Although the studies on this aspect in UHPC are relatively limited, many test results showed that PP fibers were helpful for spalling prevention of UHPC. The effect of fiber geometries and dosage on the effectiveness of spalling prevention in UHPC has also been discussed (Klingsch et al. 2013, Sun and Lin 2016, Li et al. 2019).

In a report given by Klingsch et al. (2013), PP fibers with different sizes ($\Phi 18 \mu\text{m} \times 6 \text{ mm}$, $\Phi 15.4 \mu\text{m} \times 6 \text{ mm}$, $\Phi 32 \mu\text{m} \times 6 \text{ mm}$, and $\Phi 32 \mu\text{m} \times 20 \text{ mm}$) were used to prevent spalling of UHPC. The results showed that only fibers with dimensions of $\Phi 18 \mu\text{m} \times 6 \text{ mm}$ can prevent spalling of UHPC, while other sizes of PP fibers could only minimize the extent of spalling even with a high dosage of more than 0.3 vol.%. However, the heating rate used in this study ranged from $1.5 \text{ }^\circ\text{C/min}$ to $10 \text{ }^\circ\text{C/min}$, which was relatively low. The spalling behavior of UHPC samples with PP fiber under high heating rate, e.g. ISO834 fire curve, may be different. The finding in this study only works for fibers with the size of $\Phi 18 \mu\text{m} \times 6 \text{ mm}$. The required dosages for other sizes of fibers are not provided. In addition, Sun and Lin (2016) investigated the effect of dosage and length of PP fibers on explosive spalling of UHPC under rapid heating

and rapid cooling. The experimental results indicated that 0.2 vol.% of PP fibers could provide good spalling resistance for UHPC, and PP fibers with 12mm or 19mm length performed better than the shorter ones. It should be noted that heating condition, fiber geometries and dosages used in these studies were quite different and the conclusions were not consistent.

Furthermore, Li et al. (2019) studied the effect of PP fibers on permeability of UHPC subjected to 200 °C. The investigation involved various fiber dosages and dimensions of fibers. It was found that increasing fiber dosage could not enhance permeability of UHPC when the fiber aspect ratio was less than 500. Meanwhile, when the fiber dosage was lower than 1 kg/m³, the effect of fiber aspect ratio on permeability of UHPC was negligible. The study did not involve any spalling test and did not provide any recommendations for spalling prevention of UHPC, but it clearly showed that a simplified recommendation of adding 2 kg/m³ PP fibers in Eurocode 2 was far from adequate for spalling prevention in UHPC.

In fact, there are many studies focusing on the optimization of fiber geometry and dosage for prevention spalling of HPC. Cylindrical concrete specimens with PP fibers, which had different lengths (i.e. 6 mm and 30 mm) but the same diameter (i.e. 60 µm), were heated to high temperature at a rate of 10 °C/min by Suhaendi and Horiguchi (2006). Concrete with longer fibers showed higher residual permeability than that with shorter fibers. This was attributed to the more interconnected pores inside the concrete matrix with longer fibers.

Jansson and Boström (2008) used two different diameters of fibers in their specimens. They concluded that 1 kg/m³ of PP fibers was sufficient to avoid severe spalling of concrete under standard ISO834 fire curve. The recommended fiber diameter ranged between 18 µm and 32 µm.

Bangi and Horiguchi (2012) tested the maximum pore pressure of concrete with different dimensions of PP fibers. Three sizes of PP fibers, including $\Phi 18 \mu\text{m} \times 6 \text{ mm}$, $\Phi 18 \mu\text{m} \times 12 \text{ mm}$, and $\Phi 28 \mu\text{m} \times 12 \text{ mm}$, were used. According to test results, concrete with fibers of $\Phi 18 \mu\text{m} \times 6 \text{ mm}$ and $\Phi 28 \mu\text{m} \times 12 \text{ mm}$ had the same maximum pore pressure, which was higher than concrete with fibers of $\Phi 18 \mu\text{m} \times 12 \text{ mm}$. They concluded that longer fibers with smaller diameter generally had better performance than shorter ones with larger diameter.

Heo et al. (2011) studied the effect of fiber characteristics, including diameter, length and melting point, on spalling prevention of concrete. The effectiveness of spalling protection of polyamide 6 (PA6), PP and polyvinyl alcohol (PVA) fibers with different lengths were studied by measuring the weight loss of samples subjected to 1 hour of ISO834 heating curve. A parameter NL^3k , where N is the total number of fibers, L is fiber length, and k is a factor calculated by dividing 160 to the melting point of fibers, was proposed to assess the effectiveness of fibers on spalling prevention. It was suggested that a critical threshold of $NL^3k = 190$ should be used when PP fibers were employed to prevent spalling of HSC (Heo et al. 2010). Their work is a great improvement in determining the fiber dosage for spalling prevention. However, Heo et al. (2011) did not explain the reason for choosing this parameter and the physical meaning behind this parameter was not clear. Furthermore, whether the parameter and the corresponding critical threshold could be used for UHPC has not been validated.

Maluk et al. (2017) conducted some tests to study the effect of PP fibers on spalling resistance (cube strength: around 90 MPa). The study considered different parameters of fibers, including fiber cross-section (18 or 30 μm diameter circular cross-sections and $37 \times 200 \mu\text{m}^2$ rectangular cross-section), fiber length (3, 6, 12, and 20 mm), fiber supplier (three different manufacturers), fiber type (monofilament, multifilament, or

fibrillated) and dosage (0.68-2.34 kg/m³). It was found that fiber geometry played a significant role in the effectiveness of spalling prevention. Smaller cross-section and longer fiber were beneficial for reducing spalling of concrete. Monofilament or multifilament fibers showed better performance than fibrillated PP fibers. Fibers from different suppliers did not show any difference in spalling prevention. It was also stated that the recommendation in current guidance for spalling mitigation with PP fibers was insufficient for preventing spalling of HSC. In addition, Maluk et al. (2017) also emphasized that the mechanism that was widely-used to explain the effectiveness of PP fibers in preventing spalling remained elusive and obscure.

Based on the extensive literature, addition of PP fiber is widely used for preventing spalling of UHPC. There is no doubt that it can improve spalling resistance of UHPC. The findings from HSC clearly show that fiber geometry and dosage affect the effectiveness of PP fiber in preventing spalling significantly. However, how these two parameters prevent spalling is still not clear. The experience gained from HSC may not be applicable for UHPC. In fact, the studies regarding UHPC are limited. Most of the current studies only compare several fiber geometries or fiber dosages and the conclusions are usually specific to certain geometries and dosages. There is not a recommendation that can be commonly used to determine the critical fiber geometry and fiber dosage for spalling prevention of UHPC.

In addition, to prevent spalling of UHPC, a hybrid approach of combining steel and PP fibers was also used (Pimienta et al. 2009, Pierre Pimienta 2012, Siemon and Zehfuß 2015). Test results (Siemon and Zehfuß 2015) showed that 2.25 kg/m³ of PP fibers were sufficient to prevent spalling of UHPC columns with 1-2.5 vol.% of steel fibers. In the tests conducted by Kahanji et al. (2016), a combination of 2 vol.% of steel fibers and 4 kg/m³ of PP fibers were needed to prevent spalling of UHPC beams even when the moisture content of the beam was 7.3 %. Li et al. (2019) also

confirmed that there was a synergistic effect between PP and steel fibers on spalling prevention. According to their study, adding steel fibers alone could not prevent UHPC from spalling, while adding 4 kg/m^3 of PP fibers could provide good spalling resistance. Using a hybrid of 2 kg/m^3 of PP and 157 kg/m^3 of steel fibers could eliminate spalling completely with low PP fiber dosage. A combination of PP and steel fibers could increase permeability significantly. Other recent studies showed that addition of a combination of different polymer fibers is more beneficial for prevention of spalling than a single type of fibers. This was also beneficial to maintain workability (Heo et al. 2010, Lee et al. 2012, Heo et al. 2014).

Further, Lee et al. (2012) showed that incorporating various types of fibers, including PP, PA, and steel fibers could prevent spalling of UHPC with compressive strengths ranging from 120 to 200 MPa. A total number of 36 small columns were exposed under ISO834 fire curve for 3 hours. After testing, spalling degree was evaluated by estimating the depth of spalling. Based on the experimental results, a combination of 0.4 vol.% of PP fibers with a length of 12 mm and 0.5 vol.% of steel fibers with a length of 25 mm was recommended for preventing spalling of UHPC from 120 to 150 MPa, while a hybrid mixture of PP, PA and steel fibers, with 0.2, 0.2 and 0.5 vol.%, respectively, was considered to be appropriate for minimizing spalling of UHPC over 150 MPa. In this investigation, two full-scale columns with a compressive strength of about 200 MPa were tested. The results showed that with the recommended proportion of hybrid fibers, explosive spalling could be prevented. The structural integrity of the two columns was maintained after being heated in the furnace with the ISO 834 fire curve for 3 hours.

Apart from synthetic fibers, jute fibers, which is a type of planted-based natural fibers, were shown to be effective for spalling prevention in HPC and UHPC (Ozawa et al. 2013, Ozawa and Morimoto 2014, Ozawa et al. 2019). These studies indicated that

natural fibers could be a new source for preventing spalling. However, test results on UHPC with natural fibers are still very limited. The effect of natural fibers on the properties of UHPC at high temperature needs further investigation. The mechanism of natural fibers in preventing spalling remains unclear.

2.6 Mechanism of spalling prevention with polymer fibers

2.6.1 Overview

According to previous studies, inclusion of PP fibers is an effective method to prevent spalling of concrete (Phan 2002, Noumowe 2005, Noumowe et al. 2009, Alonso et al. 2013, Ding et al. 2016). As it is one of the most widely accepted methods, this section will mainly focus on reviewing the details of PP fibers in spalling prevention, including the hypotheses on such mechanisms to prevent spalling.

2.6.2 Properties of PP fibers

Since PP fibers have a hydrophobic nature, adding them in fresh concrete will result in poor workability (Heo et al. 2012). The CTE of PP is $1.04 \times 10^{-4} / ^\circ\text{C}^{-1}$ at 23 - 80 °C (Wypych 2012) whereas CTE of concrete ranges from $0.04 \times 10^{-4} / ^\circ\text{C}^{-1}$ to $0.13 \times 10^{-4} / ^\circ\text{C}^{-1}$ at room temperature, depending on the type and content of aggregate (Neville 2011). In general, CTE of PP is approximately 8 to 26 times higher than the average CTE of concrete. Apart from normal thermal expansion, PP experiences a 7 % further expansion of volume when melting, as the semi-crystalline material is converted into lower-density amorphous material during heating (Khoury and Willoughby 2008).

The most common type of PP fibers has a melt index ranging from 0.4 g/10 min to 40 g/10 min (Wypych 2012). However, a special PP fiber, with a high melt flow rate of more than 1000 g/10 min is reported in the literature (Knack et al. 2011).

While being heated, PP fiber in concrete experiences two-phase changes with increasing temperature, as shown in Table 2.2 (Khoury and Willoughby 2008).

Table 2.2. Phase change for PP fibers (Khoury and Willoughby 2008).

No.	Temperature (°C)	Steps of phase change
1	T≈165	Temperature of melting
2	T≈200	End of melting
3	T≈325	Temperature of decomposition
4	T≈475	End of decomposition

Table 2.3. Summary of theories and hypotheses on mechanisms of PP fibers in preventing spalling of concrete.

Theory	Reference	Hypothesis
Decomposition theory	(Diederichs et al. 1995)	Void channels are generated after melting and decomposition of PP fibers occurred at high temperature.
Penetration theory	(Kalifa et al. 2001), (Knack et al. 2011)	PP fibers penetrate into surrounding cement matrix after melting, leaving microchannels.
Flowing theory	(Bošnjak et al. 2013),	Melted fibers flow into surrounding microcracks generated by thermal stresses, providing additional paths for vapor to escape.
Microcracks theory	(Khoury 2008), (Sullivan 2001), (Smith and Atkinson 2010), (Li et al. 2019)	Microcracks formed in the vicinity of fibers generate a connected network.
Interfacial Transition Zone (ITZ) theory	(Bentz 2000), (Zeiml et al. 2006)	Additional ITZs around PP fibers increase initial porosity and connect the ITZs of aggregate.
PITS theory	(Khoury 2008)	A channel between the interface of fiber and concrete paste, termed as pressure-induced tangential space (PITS), can be easily created under pressure because of poor adhesion between fiber and matrix.

2.6.3 Action of PP fibers in concrete at high temperature

Some researchers reported that adding PP fibers can significantly enhance permeability and reduce pore pressure of concrete at elevated temperature. However, the mechanism has not yet been fully understood until now. Several hypotheses, based on some experimental results, have been provided to explain the mechanism. Table 2.3 summarizes different explanations and assumptions on the action of PP fibers in concrete under high temperature. These theories will be discussed in detail in the following sections.

2.6.3.1 Decomposition theory

In the earlier years of using PP fibers to prevent spalling, it was assumed that PP fibers created void channels after decomposition (Fig. 2.10). The void channels could connect the pore networks in the cement paste and increase the permeability of the material. Nevertheless, thermal analysis shows that the decomposition of PP fibers actually starts after 300 °C (Ozawa and Morimoto 2014) (Fig. 2.11), while the increase of permeability and vapor pressure relief are commonly observed below 200 °C (Hager and Tracz 2010, Bangi and Horiguchi 2012, Bošnjak et al. 2013). In addition, it is stated that explosive spalling usually occurs in the temperature ranging from 200 °C to 300 °C (Khoury and Anderberg 2000), showing that decomposition of PP fibers has little or no effect on preventing spalling. Therefore, there must be another explanation for the beneficial action of PP fibers in concrete.

Fig. 2.10. Channel created by PP fiber at elevated temperature (Zheng et al. 2014).

Fig. 2.11. Thermal analysis of PP fibers (Ozawa and Morimoto 2014).

2.6.3.2 Penetration theory

Kalifa et al. (2001) stated that after melting at 171 °C, molten PP fibers were partially or totally absorbed by the porous network of the cement matrix, and void channels were created in the concrete. According to their observations of heated concrete using SEM, PP fibers disappeared when the temperature exceeded the fiber melting point, as shown in Fig. 2.12. A simple “water drop” test was conducted by placing a layer of fibers between two concrete surfaces (Fig. 2.13) and heating the whole set-up to a

temperature ranging between 170 °C and 200 °C with a heating rate of 1 °C/min. After the setup had cooled down to ambient temperature, the upper concrete cube was removed, and a water droplet was placed on the spot where the fiber layer was placed (Fig. 2.13). It was found that after exposure to 170 °C, the water droplet was not absorbed by the concrete sample below. Kalifa et al. (2001) stated that this was because an impermeable layer was created by molten fibers. In the set-up that was heated to 180 °C and above, the water droplet was absorbed by the concrete, but at a much slower rate than that dripped on other locations of concrete surface. Based on the test results, Kalifa et al. (2001) concluded that the melted PP fibers penetrated into the concrete and decreased the rate of water absorption.

Fig. 2.12. SEM images of PP fiber in concrete: (a) Fiber was visible before heating and (b) fiber disappeared after heated to 200 °C (Kalifa et al. 2001).

Fig. 2.13. Schematic of water droplet test by Kalifa et al. (2001).

Jansson (2013) repeated the experiment with a slight modification by using a piece of glass instead of the upper concrete cube and obtained the same results (Fig. 2.14). Moreover, PP fibers with different melt flow rates were used by Knack et al. (2011) to study the correlation between fluidity of PP fibers and spalling of concrete. The experiment showed that spalling of concrete decreased with an increase in melt flow rate. It was concluded that the diffusion of PP fibers into the concrete matrix is the mechanism that leads to creation of void channels (Knack et al. 2011).

Fig. 2.14. Water droplet test on the concrete after being heated (Jansson 2013).

On the contrary, Khoury (2008) held a standpoint that it was impossible for molten PP fibers to penetrate into cement matrix was impossible due to the high viscosity and large molecular size of PP fibers. An experiment conducted by Bošnjak et al. (2013), which will be discussed in the “flowing theory” part in Section 2.6.3.3, also indicated that absorption of PP fibers by cement paste did not happen in heated concrete. Generally, the water droplet tests by Kalifa et al. (2001) and Jansson (2013) are qualitative and the absorption of PP fibers is still questionable.

2.6.3.3 Flowing theory

In some investigations, PP fibers were found to flow into the cracks and generate additional channels for vapor pressure relief. Bošnjak et al. (2013) observed a

concrete cross-section with PP fibers before and after being heated to 200 °C using an SEM. After two days of heating at 200 °C, part of PP fibers melted and flowed into their surrounding microcracks, which in turn created voids in the initial channels (the dark portions marked with rectangles in Fig. 2.15). The observations on the samples subjected to different exposure duration at 200 °C were also discussed in their work. It was found that there was no change of PP fibers when the heating duration was 20 min, while there was some evidence to support that some portions of molten PP fibers were present in microcracks after 6 hours of heating. Although both 6 hours and 2 days seemed far too long for PP fibers to play any significant role in preventing spalling, Bošnjak et al. still concluded that the transportation of PP fibers into surrounding microcracks created additional paths in concrete, thus increasing permeability.

Fig. 2.15. SEM micrograph on concrete with PP fibers (a) before and after exposure to 200 °C for 48 hours (Bošnjak et al. 2013).

Jansson (2013) observed PP fibers flowing out of the fiber bed in a crushed specimen heated to 200 °C, as shown in Fig. 2.16. Backscattered electron microscopy analysis conducted by Liu et al. (2008) illustrated that molten PP fibers passed through their surrounding pores and left channels in concrete, resulting in an increase in connectivity of isolated pores and microcracks.

However, recent studies found that permeability of concrete starts to increase

significantly even before the melting of PP fibers occurs (Hager and Tracz 2010, Bošnjak et al. 2013, Li and Tan 2016). This cannot be explained by the flowing theory at all.

Fig. 2.16. SEM micrograph by Jansson (2013) showing molten PP flowing out of fiber bed.

2.6.3.4 Microcracks theory

Comparing the high CTE of PP fibers with that of concrete, Smith and Atkinson (2010) stated that the large difference in CTE between the fibers and the surrounding matrix could create microcracks and formed an interconnecting network, as shown in Fig. 2.17. This interconnected network of microcracks could enhance the escape of vapor (Sullivan 2001, Smith and Atkinson 2010, Li et al. 2019). However, the authors did not provide any experimental evidence to support their hypothesis.

Pistol et al. (2011) analyzed the evolution of cracks in a heated concrete sample by acoustic emission (AE) and ultrasonic measurements. The results showed that adding PP fibers led to a higher AE event and ultrasonic time-of-flight in the temperature range between 200 °C and 300 °C, which meant that PP fibers increased the formation of cracks. Micro X-ray computed tomography (CT) and SEM were also used to analyze the cracks and residual PP channels (Pistol 2016). The results confirmed that

adding PP fibers to concrete had a positive effect on formation of microcracks. The images from X-ray CT (Fig. 2.18) showed that microcracks developed at 200 °C, mainly at the region near fibers, and formed an interconnected network at 300 °C.

Fig. 2.17. Sketch on interconnecting network of microcracks generated in heated concrete (Smith and Atkinson 2010).

Fig. 2.18. Development of microcracks in heated concrete by X-ray computed tomography (Pistol et al. 2011).

However, these findings still cannot account for the increase in permeability of concrete even before PP fibers start to melt (Bošnjak et al. 2013, Li and Tan 2016). Moreover, it was not clear how the microcracks formed and propagated.

2.6.3.5 Interfacial transition zone (ITZ) theory

Bentz (2000) investigated the effect of PP fibers on explosive spalling of HPC by adopting a 3D microstructural model. Bentz proposed that PP fibers helped improve permeability of concrete by allowing percolation of ITZ in concrete. PP fibers

contributed to an increase of initial porosity of concrete by introducing additional ITZs, which were thin layers with greater porosity than cement paste (Zeiml et al. 2006). Due to PP fibers' hydrophobic nature, thin layers of water were attracted around the PP fibers during casting. As a result, a porous ITZ layer is produced around every PP fiber (Mazzucco et al. 2015), as shown in Fig. 2.19. The ITZs of fibers not only generate greater initial porosity but also help connect the ITZs around aggregates and micro air bubbles trapped in the concrete during mixing. These effects, in turn, increase the initial porosity of concrete at room temperature.

Fig. 2.20 illustrates a schematic diagram showing the connection of ITZs around aggregates by PP fibers. A coupled hygro-thermal-mechanical finite element model incorporating this effect has been conducted by Mazzucco et al. (2015). The model was used to analyze evolution of pore pressure in concrete samples and the predictions compared reasonably well with test results.

Nonetheless, this theory has not been experimentally confirmed yet. In contrast, some experimental results showed that PP fibers did not significantly affect permeability values of HPC and UHPC at ambient temperature (Liu et al. 2008, Bošnjak et al. 2013, Li et al. 2018). Furthermore, no distinct porosity layer has been identified in the ITZ of UHPC (Wang et al. 2012), indicating that this theory may not be suitable for UHPC.

Fig. 2.19. ITZ around PP fibers (Mazzucco et al. 2015).

Fig. 2.20. ITZ connection due to PP fibers (Mazzucco et al. 2015).

2.6.3.6 Pressure-induced tangential space (PITS) theory

Khoury (2008) believed that PITS can be generated before melting of PP fibers. He considered that due to PP fibers' hydrophobicity, interfacial adhesion between the fiber and the concrete matrix was so poor that it could be easily disrupted by vapor pressure. When the temperature of concrete was higher than 100 °C, high vapor pressure generated in the concrete could overcome the expansion of the fiber and rupture the weak interfacial contact between the fiber and the cement matrix. Therefore, a tangential space around the PP fiber was formed at the interface of the fiber and the cement paste. Fig. 2.21 illustrates the concept of vapor pressure passing through the PITS of a PP fiber. The effectiveness of such mechanism is dependent on the surface area and connectivity of fibers.

Fig. 2.21. Formation of PITS (Khoury 2008).

Although this theory was accepted by some researchers (Bošnjak et al. 2013, Ozawa

and Morimoto 2014), it has not yet been supported by any experimental evidence. Smith and Atkinson (2010) considered the dispersed distribution of PP fibers in concrete and argued that neither of the fiber connectivity and pressure alleviation could be clearly explained in Khoury's theory.

In addition, Knack et al. (2011) also mentioned that this mechanism did not take place in heated concrete. A special type of highly cross-linked polyethylene (PE) fiber, which is also hydrophobic, was added to concrete with the expectation of generating PITS during heating period. However, the results showed that the amount of spalling in concrete with these special PE fibers was twice that of specimens without fibers. The authors concluded that PITS did not occur.

Till now, the PITS theory by Khoury (2008) remains a hypothesis. Although some researchers cited it to explain their own test results, there is still a lack of test evidence to support the presence of PITS.

2.6.3.7 Summary on mechanism of spalling prevention

In this section, a critical review of different hypotheses on the mechanism of PP fibers in spalling prevention is presented. It was found that these theories did not have experimental evidence or were incongruent with the PP fiber properties. More importantly, some widely accepted test results could not be explained by the proposed mechanisms. Furthermore, most of the studies mainly focused on NSC or HSC. For UHPC, which has a much denser microstructure and no ITZ, the exact role of PP fibers in preventing spalling is still not fully understood.

2.7 Summary

In this chapter, mechanical properties of UHPC and microstructural changes of

concrete at high temperature are thoroughly reviewed. UHPC is susceptible to explosive spalling when exposed to fire. Explosive spalling, in turn, reduces the cross-sections of structural members and increases the heat flow rate to the inner layers of reinforcement, resulting in a reduction on load-carrying capacity of structures. A comprehensive review of previous works suggested that the spalling of concrete is caused by thermal stress and/or build-up of vapor pressure. Inclusion of polymer fibers, such as PP fibers, is one of the most commonly used methods to prevent spalling. Although quite a number of mechanisms of spalling prevention with PP fibers have been proposed in the literature, many of them remain as hypotheses and are not yet validated by experimental results. Some hypotheses are even in conflict with test observations. Meanwhile, many other types of polymer fibers were used for spalling prevention, but there are limited conclusions that can be used to guide the fiber selection. The properties needed for polymer fiber in spalling prevention remain unanswered. Therefore, it is necessary to re-examine the behavior of polymer fibers in UHPC and to clarify the mechanism of spalling prevention. This will be the main objective of CHAPTER 3.

Many studies have been conducted to optimize the dosage and geometry of PP fibers for spalling prevention. Based on the extensive literature, fibers with various geometries have been adopted for preventing spalling. The diameter varies from 15 μm to 200 μm (Lee et al. 2012, Ozawa et al. 2014), while the length varies from 4 mm to 30 mm (Heo et al. 2010, Ozawa and Morimoto 2014). Meanwhile, the content of fibers also varies from 1 kg/m^3 to 12 kg/m^3 (Klingsch et al. 2013, Xiong and Liew 2015, Li et al. 2019). According to the literature, 2 kg/m^3 of PP fibers, which is recommended in Eurocode 2, is inadequate for preventing spalling of UHPC. It is also found that fiber geometry and dosage have a high effect on permeability and spalling prevention of concrete. However, the recommendations on these two parameters for preventing spalling of UHPC in the literature are rather different and

are very limited. Most of the studies only focused on very limited types of fiber geometries and dosages, resulting in different findings. In addition, despite voluminous publications on spalling, the mechanisms of fiber geometry/dosage affecting spalling resistance remain elusive and unclear. This author believes that fiber distribution and connectivity are important factors that affect spalling resistance. However, there is only limited experimental data on this matter. The effect of fiber geometry and dosage on fiber distribution and connectivity need to be carefully studied in detail. The critical geometry and dosage of PP fibers for preventing spalling of UHPC also need to be further investigated. CHAPTER 4 and CHAPTER 5 of this research work will focus on these two aspects.

Furthermore, although natural fibers are incorporated in concrete for spalling prevention, only preliminary data is available. As the thermal properties of natural fiber are different from polymer fibers, a thorough investigation on the effect of natural fibers on spalling prevention can enhance the understanding on spalling prevention. There is also limited information available on how natural fibers affect the concrete properties at high temperature and durability. It is necessary to conduct a thorough study on the topic of high temperature performance of UHPC with natural fibers. This will be investigated in CHAPTER 6.

CHAPTER 3 MECHANISM OF SPALLING PREVENTION IN UHPC

3.1 Overview

According to the literature, many spalling prevention mechanisms with PP fibers (added to concrete in the range of 1 to 3 kg/m³) were proposed. Nonetheless, melting of PP fibers and creation of pathways to relieve the vapor pressure built-up in concrete is the most commonly accepted spalling resistance mechanism in the literature (Dwaikat and Kodur 2010, Ozawa and Morimoto 2014). Despite the extensive literature on this topic, many aspects of this proposed mechanism are qualitative and sometimes even speculative, and many questions still remain unanswered. For instance,

- (i) What is the role of molten polymer in spalling (which generally happens in the temperature range of 200-300 °C (Khoury 2000))?
- (ii) How does this polymer melt flow in concrete so as to create empty channels to release trapped vapor pressure as the polymer does not even start to decompose until 265 °C (onset decomposition temperature, T_{5%}, where 5 % of mass loss occurs)?

Another important aspect is the currently proposed hypotheses cannot explain some test results reported recently.

In this regard, the major focus of this chapter is to thoroughly re-examine the exact role of PP fibers in the prevention of explosive spalling of UHPC. For this purpose, a range of analytical, physical and microscopic characterization techniques are used

on the concrete samples before and after subjecting to elevated temperature.

3.2 Design of experiment

3.2.1 Material and sample preparation

The mix proportion of materials used for UHPC in this study is shown in Table 3.1. The mean size of silica sand was 110 μm . Natural river sand with a maximum size of 0.6 mm was used as fine aggregate. PP fibers with a density of 910 kg/m^3 and an aspect ratio of ~ 360 (12 mm length and 33 μm diameter) were obtained commercially. Polycarboxylic type superplasticizer (Sika ViscoCrete-2044) was used in all concrete mixtures. Samples of UHPC with and without PP fibers are denoted as UHPC/F and UHPC/O, respectively, hereafter. The specimens were demolded after casting for 1 day and stored in lime-saturated water at ambient temperature for 27 days. The compressive strength values of UHPC with and without PP fibers at 28 days are $\sim 148.6 \pm 3.0$ MPa and 159.9 ± 2.9 MPa, respectively.

Table 3.1. Mixture compositions of UHPC with and without PP fibers (unit in kg/m^3).

Mixtures	W/B ^a	Cement ^b	Silica fume	Silica sand	Superplasticizer	Fine Aggregate	Water	PP fiber
UHPC/O	0.2	830	208	208	33	913	208	0
UHPC/F	0.2	830	208	208	33	913	208	3

^a W/B: water to binder ratio; ^b Portland cement (ASIA@CEM II 52.5 R).

The samples for permeability test were cast in disc-shaped molds with 45 mm in depth and 150 mm in diameter. After curing, the discs were grinded on both faces and smoothed, with a final thickness of ~ 40 mm. Cylinder-shaped samples with 50 mm diameter and 100 mm height were also prepared for spalling tests at elevated

temperature.

3.2.2 Thermal analysis

Differential scanning calorimeter (DSC, from TA Instruments, model: Q10) and thermogravimetric analysis (TGA, from TA Instruments, model: Q500) were conducted to understand the melting and decomposition behavior of PP fibers and concrete mix. A constant heating rate of 5 °C/min from room temperature to 400 °C was employed in DSC, while in TGA, a ramp of 5 °C/min was used to heat up to 400 °C in the air atmosphere. The gas flow used in both DSC and TGA was set at 100 ml/min. The weight of samples of UHPC/O and UHPC/F in TGA was 27.251 mg and 30.275 mg, respectively. To understand the dimensional changes and variations in coefficient of thermal expansion (CTE) of fibers, cement paste, and aggregates with temperature, thermomechanical analyzer (TMA, from TA Instruments, model: Q400) was employed. The samples were heated in a furnace with a probe in contact that is connected to a strain detector and a force generator. The force was set at 0.01 N. For the cement paste and aggregate, the samples were heated to 300 °C with a heating rate of 5 °C/min, while the maximum temperature of 150 °C was used for PP fibers. All the TMA tests were performed under nitrogen atmosphere. The set-up of TMA is shown in Fig. 3.1.

Fig. 3.1. Set-up of TMA.

3.2.3 X-ray diffraction

X-ray diffraction (XRD) measurements using CuK α radiation were conducted on UHPC/O and UHPC/F to identify the crystal phases. The sample sections were 18 mm in length, 12 mm in width and 3 mm in thickness. Four batches of samples were prepared for each mix for testing at different selected temperatures, i.e., ambient, 105, 150, and 250 °C. Since spalling of concrete usually occurs at a temperature ranging from 200 °C to 300 °C (Khoury 2000, Klingsch 2014), only behaviors under 300 °C are of interest in this study.

3.2.4 SEM observation

A scanning electron microscope (SEM, JSM 6360) operating at an accelerating voltage of 5 kV was used for observing microstructural changes of UHPC/F and UHPC/O at different temperatures after carefully preparing the samples following a series of grinding and polishing steps. 800, 1200 and 2500 grit sandpapers were used for initial grinding of the samples for 0.5, 2 and 10 min, respectively. Then, the samples were polished on a Buehker TexMet P cloth (hard perforated and non-woven) by using 1 μ m diamond paste for 20 min. Afterwards, the samples were rinsed for 5 min with isopropanol in an ultrasonic bath. For each target temperature (chosen as 105, 150, 170, 200 and 300 °C), to obtain information before and after melting of fibers, samples were kept in a convection furnace for approximately 30 min. Subsequently, they were allowed to cool down naturally in the furnace.

To observe a representative area/number of fibers at high resolution (for identifying microcracks in samples) in an SEM micrograph, 25 micrographs at a fixed magnification (500x, 0.2 μ m/pixel, covering an area of 256 $\times$ 192 μ m²) were taken and stitched together. This selection was based on the findings in (Ye 2003, Wong et al.

2006) where it was noted that 20 frames of images at a magnification of 500x were necessary for meaningful image analysis. In this study, micrographs were obtained at three random locations in each sample. To perform an accurate fully automated image analysis for quantification of microcracks, it is a requirement that the original image is of exceptional resolution with good contrast and has sharp transitions from white to black (and vice versa). Generally, SEM micrographs of concrete do not possess these qualities, and therefore, automated detection and crack segmentation methods are usually unreliable. Hence, a semi-automated approach was adopted here. The stitched SEM micrograph was converted to a binary image in which the pixels of microcracks were set to 1 (black) and the pixels of background were defined as 0 (white) by using Fuji/ImageJ software, developed by National Institutes of Health (NIH) (Abràmoff et al. 2004). This was followed by manual tracing using Photoshop. Finally, image analysis was performed. Based on isotropy hypothesis of crack distribution and statistics for crack length, stereology parameters were extracted from 2D images to describe the networks of cracks (Zhou et al. 2012, Wu et al. 2015, Wang et al. 2016). In this study, number density of microcracks (total number of microcracks divided by the image area), crack area fraction (% of crack area to the image area) and crack connectivity were quantified. Crack network (connectivity), ϕ , is defined by the following equation (Zhou et al. 2012):

$$\phi = 1 - \frac{\sum_{j=1}^m l_j^2}{\sum_{k=1}^n l_k^2} \quad (3-1)$$

where m and n represent the number of isolated and total number of microcracks, respectively. l_j is the respective length of the j^{th} isolated microcrack and l_k is the respective length of the k^{th} microcrack.

3.2.5 Gas permeability measurement

Both residual and hot permeability values of UHPC/O and UHPC/F were measured. In permeability test, samples were heated by using an electric furnace with a heating rate of 1 °C/min to all the selected temperatures, followed by maintaining at the target temperature until a constant mass (equilibrium) was reached to exclude the effect of moisture. The low heating rate used in permeability tests was to avoid undesirable temperature gradients and thermal stresses developed inside the specimens (Li et al. 2018, Li et al. 2019). Afterward, samples were naturally cooled down to about 60 °C in the furnace. Subsequently, samples were sealed in a desiccator containing silica gel and naturally cooled down to room temperature. After each heat treatment, residual permeability of the specimen was measured using Torrent permeability tester, which was a standard handheld permeability device developed by Torrent (1992) and was further analyzed for general applications by Jacobs and Hunkeler (2006). Three specimens of each concrete mix were employed in the test and the average values were reported here. Additionally, hot permeability of UHPC/O and UHPC/F samples was also measured at the same target temperature. At least two specimens of each concrete mix were tested. Details of the test setup and procedure for evaluating hot permeability are given in (Li and Tan 2016, Li et al. 2018).

3.2.6 Spalling test

Spalling tests were conducted to show the effectiveness of PP fibers in preventing spalling of UHPC. Three cylinders with 50 mm diameter and 100 mm height were tested for each mix. The cylinders were heated in an electrical furnace for 1 h in accordance with the standard ISO 834 heating curve, as shown in Fig. 3.2. Afterwards, the cylinders were allowed to cool down naturally in the furnace for 24 h. Then the extent of spalling was measured and documented.

Fig. 3.2. Heating curve of spalling test.

A summary of the tests conducted in this investigation is shown in Table 3.2.

Table 3.2. Summary of tests.

Sample	Tests
Concrete (UHPC/F and UHPC/O)	Residual permeability
	Hot permeability
	Spalling test
	DSC, TGA, XRD
	SEM
PP fiber	TGA, DSC,
Cement paste, aggregate, PP fiber	TMA

3.3 Result and discussion

3.3.1 Spalling behavior and the extent of gas permeability

As evident in Fig. 3.3, the presence of PP fibers in UHPC mitigated explosive spalling when exposed to ISO 834 heating curve. However, the samples without PP fibers exhibited severe spalling with the entire specimen breaking into small pieces. As a first step to explain these differences, both residual and hot permeability values of

UHPC with and without fibers were measured as a function of temperature (Fig. 3.4). Due to the dense microstructure of UHPC, it was difficult to obtain gas permeability of UHPC at ambient temperature. Therefore, UHPC was dried at 105 °C before measuring the permeability, in line with other reported studies (Wu et al. 2015, Li and Tan 2016). In general, the gas permeability increased with increasing temperature for both UHPC/O and UHPC/F. Also, in the presence of fibers, at any measured temperature, residual permeability of UHPC/F was always larger than UHPC/O. More importantly, permeability of UHPC/F showed a significant increase by two orders of magnitude between 105 °C and 150 °C. The permeability further increased by one order of magnitude between 150 °C and 200 °C. With a further increase in temperature beyond 200 °C, the rate of increase in gas permeability of UHPC/F is less. Furthermore, it should be noted that despite the quantitative differences between residual and hot permeability data, both follow a very similar trend. The quantitative differences are expected as there are fundamental variations between residual and hot permeability techniques. For example, residual permeability values are based on air viscosity, whereas hot permeability values are dependent on vapor viscosity, and their respective mobility through the network of cracks in the concrete sample. There are other factors that could influence the differences in quantitative data obtained from these two techniques like the closure of cracks during cooling down, recrystallization/solidification of PP, and the differences in testing methodologies by themselves. However, to what extent these different factors are contributing to the differences between hot and residual permeability is beyond the scope of this study, and the authors will investigate this aspect in detail in their future work. In the current work, the intention is to show that a similar trend of permeability (especially, the increase in permeability of UHPC/F at 150 °C) is observed irrespective of the technique.

The permeability test results of UHPC/F (Fig. 3.4) are extremely interesting as a

significant increase of two orders of magnitude was observed even before reaching the melting point of PP fibers (which is $\sim 170^\circ\text{C}$) (Fig. 3.5). This clearly suggests the failure of many theories that have been proposed to account for the improvement in spalling resistance of UHPC with PP fibers. It is also evident from Fig. 3.5 that the decomposition onset of PP fibers is well beyond 250°C , and at 350°C the mass loss is almost 97 %, which again illustrates the failure of ‘melting of PP fibers and creation of empty channels’ theory for relieving the vapor pressure developed in the concrete. Further, XRD spectra of UHPC/O and UHPC/F subjected to different temperatures are similar qualitatively (in terms of peak positions) with no obvious differences (Fig. 3.6). This clearly indicates the absence of any chemical/phase changes in UHPC until 250°C .

Fig. 3.3. Results of spalling test for (a) UHPC without fibers, and (b) UHPC with fibers subjected to standard ISO 834 heating curve for 1 hour.

Fig. 3.4. Residual and hot gas permeability of UHPC with and without fibers and with increasing temperature.

Fig. 3.5. TGA-DSC data of PP fibers used in the current study.

Findings from XRD data is also supported by the DSC and TGA data of UHPC/O and UHPC/F (Fig. 3.7), which suggest the absence of phase changes of UHPC until 250 °C. As evident, below 200 °C, there are only peaks related to evaporation of free water. Subsequently, at around 250 °C, dehydration of calcium silicate hydrate (C-S-H) phase causes the loss of bound water (Noumowe 2005). In short, XRD and DSC-TGA data of UHPC/O and UHPC/F suggests that the changes in permeability cannot

be attributed to any of the phase/chemical changes of concrete.

Fig. 3.6. XRD spectra of (a) UHPC/O and (b) UHPC/F subjected to different temperatures. C_2S is dicalcium silicate, C_3S tricalcium silicate, and CH calcium hydroxide.

Fig. 3.7. TGA, DTG and DSC results of UHPC/O and UHPC/F.

3.3.2 Dimensional changes with temperature

As explained in section 3.3.1, considering the size (12 mm in length and 33 μm diameter) and number density of PP fibers in the concrete matrix ($3.2 \times 10^8/\text{m}^3$), the significant increase in permeability between 100 °C and 150 °C cannot be explained by any of the mechanisms proposed in the literature. The authors believe that melting of fibers is not related at all to the increase in permeability of concrete matrix. The difference in coefficient of thermal expansion or in other words mismatch of thermal expansion between the polymer and the concrete is the key parameter. To explain this further, dimensional changes with increasing temperature of cement paste, aggregate and PP fibers are shown in Fig. 3.8. Clearly, PP fibers show relatively much larger dimensional changes than cement paste when exposed to high temperature. At 105 °C, the relative dimensional change of PP fibers is almost 41 times larger than that of cement paste, and it further increases at 150 °C. This provides the first clue of what exactly happens even before melting of fibers. The huge difference in CTE of PP and cement paste is capable of resulting in the formation of microcracks at the interface

of PP fibers. This relieves the build-up of internal stresses through the formation of a permeable transport system for escaping water vapor (permeation effect) if the number density of fibers is high enough to create an interconnected network of cracks.

Fig. 3.8. Dimensional (strain) changes of PP, cement paste and aggregate with increasing temperature. As can be seen, the dimensions of PP increased significantly with temperature. Based on these experiments, CTE of PP is $1.367 \times 10^{-4} / ^\circ\text{C}$ below 80°C , $3.896 \times 10^{-4} / ^\circ\text{C}$ between 80°C and 105°C , and $2.084 \times 10^{-3} / ^\circ\text{C}$ between 105°C and 150°C .

In fact, the phenomenon of mismatch of CTEs is widely reported in the composites field (for example, see (Folias and Hohn 1998)). The mismatch between the CTE of the matrix and the fibers could result in stresses (during cooling from high temperatures) that are often sufficient to produce microcracking even in the absence of external loads. Cracks are reported to develop at the interface of fibers and matrix at 45° and then advance into the matrix until they reach adjacent fibers. The relative dimensional changes between cement paste and aggregates at higher temperatures ($> 160^\circ\text{C}$ and up to 300°C) could also result in microcracks originating at the interface of aggregates. Therefore, it is expected that even the aggregate size and volume

fraction will influence the formation of microcracks induced by aggregate restrained shrinkage (due to differences in expansion/shrinkage coefficient mismatch between aggregate and concrete). However, considering the aspect ratio of the aggregate in the present system (~ 1.2), the contribution of this to the overall network of microcracks is expected to be small.

3.3.3 Microscopic investigation of the role of PP fibers

To understand the microstructural evolution in UHPC/F with changes in temperature, a representative area containing a few fibers was chosen and exposed to different temperatures. An SEM micrograph of this representative region at different temperatures is shown in Fig. 3.9. At ambient temperature, there are no obvious cracks around the fibers and fine aggregates (0.6 mm) (Fig. 3.9a). After exposing the sample to 105 °C, some microcracks appear around the PP fibers, as shown in Fig. 3.9b. This is a result of mismatch in expansion coefficients of PP fibers and matrix or in other words, caused by restrained expansion of PP fibers. According to Fig. 3.8, the dimension change of PP fibers at 105 °C in unconstrained conditions is already 25 times than that of cement paste. Though the fibers are not completely debonded, Fig. 3.9b clearly reveals that the formation of microcracks starts at the interface in the radial direction.

If loading was to be applied on these samples along with rising temperature, then there may be further widening of microcracks or even closing depending on the type of imposed loading. Here, temperature is the only variable and changes in microstructure (and thus, permeability) can be directly related to it. At 150 °C, the size and length of cracks increase along with the appearance of new radial cracks from fibers to matrix. These microcracks are found to link those created by the neighboring fibers and form an interconnecting network, as shown in Fig. 3.9c.

Moreover, the inset in Fig. 3.9c shows a higher magnification SEM micrograph of the PP fiber interface. It clearly points to debonding of fibers from the matrix. This reiterates that thermal stresses caused by the mismatch in thermal expansion coefficients of the fibers and matrix phases are large enough to cause microcracking of the matrix.

Similar microcracks are also observed as a result of aggregate restrained shrinkage in (Wu et al. 2015) and (Grassl et al. 2010). Lu et al. (1991) have characterized the matrix cracking in brittle matrix composites induced by mismatch of thermal expansion coefficients between the matrix and the fiber by using a non-dimensional cracking coefficient. The magnitude of this coefficient was governed by incompatible strain, matrix toughness and modulus, fiber size and volume fraction. In these matrices, it was noted that interfacial debonding encouraged matrix cracking and that the cracks extended radially between the fibers. In the current test specimens, as the PP fiber has a higher CTE than the matrix, the matrix is in compression and should, in fact, provide resistance to matrix crack opening. However, the occurrence of debonding at the interface and matrix cracking suggests that the relative difference in CTE is too large. Besides, the cement paste shrinks at higher temperatures.

Fig. 3.9. Microstructural evolution in UHPC/F sample with temperature: (a) ambient temperature, (b) 105 °C, (c) 150 °C, (d) 170 °C, (e) 200 °C, and (f) 300°C.

Therefore, it can be concluded that the network of cracks formed throughout the matrix initiated from the fiber-matrix interface is the primary reason for the huge increase in gas permeability at temperatures less than 170 °C. This is also evident from the calculations of crack density, crack connectivity and crack area fraction in the samples (Fig. 3.10), which show an increase from 105 °C to 150 °C and 170 °C. The crack connectivity, in particular, is an important (geometrical) factor governing gas permeability through concrete (Ringot and Bascoul 2001). At 170 °C, which is the peak of the melting endotherm of PP fiber, some PP fibers disappeared partially from their channels (Fig. 3.9d). Nevertheless, new microcracks are also observed at 170 °C, and more importantly, the existing microcracks widened and grew in length and in width. As the onset decomposition of PP fibers is around 265 °C, this suggests that the molten polymer might have flown into surrounding cracks. However, absorption of melt by surrounding matrix is not possible due to the highly viscous nature of the melt. In fact, the authors have carried out a simple experiment on this aspect where a few PP fibers were sandwiched between a glass slide and UHPC. The setup was heated to 200 °C and maintained at that temperature for one hour. After cooling down from heating, cross-sections at different places were cut and checked for the absorption of melted PP fibers into concrete. Little evidence of molten PP penetrating into UHPC was found.

At higher temperatures of 200 °C and 300 °C (Fig. 3.9e and f), most of the PP fibers have disappeared. Importantly, the disappearance of fibers and the creation of empty channels did not contribute significantly to the permeability of concrete at these temperatures (Fig. 3.4). This is despite an increase in the total crack area fraction due to the empty channels left behind by PP fibers (Fig. 3.10c). This reiterates the importance of creation of interconnected network of cracks at temperatures lower than the melting point of fibers.

Fig. 3.10. Changes in (a) number density, (b) inter-connectivity, and (c) area fraction of microcracks with temperature. The dotted line in (c) considered the areas left by melted fibers.

Fig. 3.11. Microstructural evolution in UHPC/O sample with temperature: (a) ambient temperature, (b) 105 °C, (c) 150 °C, (d) 200 °C, and (e) 300 °C.

In contrast, in the absence of fibers, the number of microcracks formed in UHPC sample is small even at a high temperature of 200 °C. An example of this behavior is shown in Fig. 3.11, which clearly indicates the absence of interconnected network of cracks. This is expected considering the size and number density of aggregates, as well as the relatively small thermal mismatch between aggregates and cement paste (Fig. 3.8). This ultimately resulted in limited number of microcracks extending from the interface of aggregates into concrete matrix. Essentially, the permeability of UHPC/O at temperature of 200 °C remained at a relatively low level.

To further confirm the mechanism of polymer fibers in preventing spalling of UHPC, some other types of polymer fibers, including linear low-density polyethylene (LLDPE), ultra-high molecular weight polyethylene (UHMWPE), polyamide 66 (PA66) and polyester (PET) fibers were added in UHPC. The test results (as reported in APPENDIX A) clearly showed that polymer fibers with high CTE showed better performance on spalling prevention of UHPC.

Nonetheless, it must be highlighted that the crack network in these samples is expected to be three-dimensional, and thus, the increase in inter-connectivity (Fig. 3.10b) is even more significant. The observations presented in Fig. 3.9 and Fig. 3.11 and the associated crack calculations in Fig. 3.10 are based on two-dimensional analysis only. A further investigation by using 3D X-ray tomography would be excellent to support the important findings. But the required high resolution of tomography versus the size of sample may compromise the scale of observations, as the recommended sample size is 1000 to 2000 times of the resolution in X-ray tomography (Bossa et al. 2015). To achieve a submicron resolution, the sample size should be less than 1 mm.

In summary, this work sheds light on the mechanism of prevention of explosive

spalling in concrete with polymer fibers. The results presented here show that the previously proposed mechanism of melting of polymer fibers in enhancing the permeability of gases through concrete may not be a correct reflection of the actual behavior observed. The thermal mismatch between the fibers and the matrix is critical to obtaining an interconnected network of cracks in the matrix even before melting of fibers. In fact, these observations suggest that polymer fibers per se are not required and even other choices of materials may be able to increase the permeability of concrete. The requirements for the materials are: (i) exhibit a huge thermal mismatch with the cement matrix, and (ii) high aspect ratio of the fibers. The higher the aspect ratio of the fibers, the greater the number density of these dispersed particles in the matrix, the higher the crack density and interconnectivity will be. More importantly, this work provides the foundation and solutions for resisting explosive spalling in concrete with compressive strength of greater than 150 MPa. This means by simply focusing on the phase boundary between the polymer fibers and the matrix, it is possible to tackle spalling by initiating a network of fine cracks upon an increase in temperature.

3.4 Summary

This chapter re-examines the mechanism of preventing spalling of UHPC by polymer fibers. Based on the experimental results, it is confirmed that polymer fibers improve the spalling resistance of UHPC by increasing the gas permeability with temperature. The permeability of UHPC showed a two-orders increase in magnitude when exposed to 150 °C, which is below the melting point of PP fibers. The permeability further increased by another order of magnitude between 150 and 200 °C. With a further increase in temperature (to 200 °C and even 300 °C), the rate of increase in gas permeability in the presence of fibers is relatively small. This suggests that the empty channels (of PP fibers) left behind do not contribute significantly to the permeability

of concrete. This reiterated the importance of the creation of interconnected network of cracks at temperatures lower than the melting point of fibers. The formation of microcracks is a result of a huge mismatch in thermal expansion coefficients of the matrix and the fibers. This is confirmed by TMA and supported by SEM observations. Furthermore, XRD and TGA-DSC results of concrete with and without fibers also confirmed the absence of any chemical/phase changes of UHPC at temperatures less than 250 °C, which is the critical range for permeability measurements.

CHAPTER 4 SPATIAL DISTRIBUTION OF PP FIBERS IN UHPC

4.1 Overview

Based on the work in CHAPTER 3, it was concluded that the interconnected network of cracks caused by relative thermal expansion differences between PP fibers and cement matrix is responsible for enhancing permeability. This reduces the susceptibility of explosive spalling of UHPC. Therefore, the distance between the fibers and their connectivity in UHPC are critical parameters for forming the interconnecting network. In fact, there is always a concern on how fibers or microcracks are connected with one another and the spatial distance between fibers (Smith and Atkinson 2010). However, most of the previous investigations are based on 2-dimensional (2D) SEM images (Zeiml et al. 2006, Liu et al. 2008). As SEM images can only provide information on the planar morphology, it is impossible to characterize spatial fiber distribution, which is in 3-dimensional (3D) (Kim et al. 2013, Bordelon and Roesler 2014).

In this chapter, X-ray computed tomography (X-ray CT) is adopted to obtain spatial distribution of polymer fibers. The effect of fiber distribution in 3D space in UHPC on spalling resistance is investigated. For this purpose, spalling tests and permeability tests are conducted on UHPC samples with different dosages of PP fibers. In addition, flowability and compressive strength are also obtained to study the effect of fibers on mechanical properties of UHPC.

4.2 Material and experimental procedure

The mix proportion used was the same as that mentioned in CHAPTER 3. PP fibers (from Dingqiang, China) with different geometries and dosages were added to UHPC, as shown in Table 4.1. For the control group without any fibers, it was named as UHPC-C, while the other mixes were named after diameter, length, and dosage of fibers. For example, U33/06/03 meant that PP fibers in this mix were 33 μm in diameter and 6 mm in length with a fiber dosage of 3 kg/m^3 .

Sample preparation and curing followed the same procedure mentioned in CHAPTER 3. Cubic (50 mm $\times$ 50 mm $\times$ 50 mm), cylindrical (Φ 50 mm $\times$ 100 mm), and cylindrical disc (Φ 150 mm $\times$ 48 mm) molds were used to cast the samples for compressive strength tests, spalling tests, and permeability tests, respectively.

Table 4.1. Mixture compositions.

Mix design	Relative weight ratio to cement						PP fiber (kg/m^3)	Fiber dimension			Fiber number density ($/\text{mm}^3$)
	C	SS	SF	FA	SP	W/B		Diameter (μm)	Length (mm)	L/D	
UHPC-C	1.0	0.25	0.25	1.1	0.04	0.2	0.0	-	-	-	0
U33/06/03	1.0	0.25	0.25	1.1	0.04	0.2	3.0	33	6	182	0.643
U33/12/03	1.0	0.25	0.25	1.1	0.04	0.2	3.0	33	12	364	0.322
U100/06/03	1.0	0.25	0.25	1.1	0.04	0.2	3.0	100*	6	60	0.070
U100/12/03	1.0	0.25	0.25	1.1	0.04	0.2	3.0	100*	12	120	0.035
U100/12/06	1.0	0.25	0.25	1.1	0.04	0.2	6.0	100*	12	120	0.070
U100/12/09	1.0	0.25	0.25	1.1	0.04	0.2	9.0	100*	12	120	0.105
U100/12/12	1.0	0.25	0.25	1.1	0.04	0.2	12.0	100*	12	120	0.140

C: cement, SS: silica sand, SF: silica Fume, FG: fine aggregates, SP: superplasticizer, W/B: water-to-binder ratio,

L/D: aspect (length to diameter) ratio.

*Diameter varies from 90 to 110 μm , with an average of about 100 μm .

4.3 Material properties

Flow of fresh UHPC mixtures (ASTM 2015) was tested according to ASTM C1437-15. The tests were conducted right after the mixing process. Compressive strength tests were conducted according to ASTM C109/109M-16a (ASTM 2016). A hydraulic compression machine with a capacity of 3000 kN was used for the tests. During the test, a constant load of 100 kN/min was adopted, and the maximum force was recorded. The compressive strength tests were conducted on the UHPC samples at the age of 7 and 28 days, respectively. Three samples were used for each test. Spalling tests and permeability tests followed the same procedure mentioned in CHAPTER 3.

4.4 X-ray CT measurement

4.4.1 Acquisition of CT images

In this study, a high energy micro-CT scanner (Bruker SkyScan 1173) was used to conduct X-ray CT scanning of UHPC samples. A cylindrical sample was recommended for X-ray CT scanning to minimize computer reconstructed artifacts produced from any non-uniform specimen thickness (Hsieh 2009, Bordelon and Roesler 2014). It was also suggested that more than 4 pixels were needed to identify an object or a component effectively and reliably in the X-ray CT reconstructed model. The recommended sample size was about 1000 to 2000 times the resolution in X-ray tomography (Hsieh 2009, Yio et al. 2015). As the PP fibers used in this study had a minimum diameter of 33 μm , a small sample with a diameter of around 14 mm was chosen to reach a compromise between the required resolution and the size of sample. As a result, small cylindrical samples ($\Phi 14 \text{ mm} \times 30 \text{ mm}$) were cored from UHPC block of each mix. For an X-ray CT system, a higher penetrating power requires a

higher scanning voltage. In addition, a higher voltage could increase the spatial resolution and in contrast, decrease the functioning stability of CT scanner (Wang et al. 2018). The X-ray attenuation of PP fibers and air bubbles are similar in magnitude but different from the cement matrix. Therefore, some preliminary tests were conducted to determine a suitable energy setting, which was high enough to distinguish fibers and voids in the cement matrix without oversaturating the detector of the X-ray CT system. After the iteration, 130 kV of source voltage and 60 μ A of source current were employed to generate a pixel resolution of 7.8 μ m so that individual fibers could be observed. After the CT system was ready, the UHPC sample was fixed vertically on the rotation table. Then the sample was rotated with a rotation step of 0.2 ° during scanning. The sample and X-ray CT system are shown in Fig. 4.1.

Fig. 4.1. X-ray CT system

4.4.2 Imaging processing

Image enhancement was carried out to improve contrast resolution and alter brightness in every original 2D image. Due to different energy attenuation of X-ray, PP fibers and air bubbles were represented by black color, while cement matrix and aggregates were represented by grey in color in a 2D image. Therefore, threshold and

segmentation processes (Abràmoff et al. 2004) were applied to simplify the analysis by removing all cement matrix and aggregates. After filtering through the threshold, the original images were converted to binary images, in which air bubbles and PP fibers were in black, and the other parts were in white.

To avoid the effect of beam-hardening, which causes the edges of an object to appear brighter than the center, an area of 900 pixels×900 pixels was cropped from the central part of the images for further analysis, as shown in Fig. 4.2.

Fig. 4.2. Image acquisition of CT images.

Due to similar pixel intensity caused by similar X-ray attenuation, it was not possible to separate PP fibers from air bubbles by using intense pattern recognition functions. Since it was unlikely for fibers to be aligned in one unique plane, a 3D processing method, including shape and size filtering, was adopted in the literature to differentiate between fibers and air bubbles in CT images (Bordelon and Roesler 2014). In a 3D-reconstructed model based on the threshold images, the sphericity (Bordelon and Roesler 2014) and Feret shape (length to width ratio) of each object were computed. Then objects with high sphericity and low Feret shape were defined as air bubbles. This way, fibers, and air bubbles could be separately identified. In this

process, some other image processing techniques, including opening, subtracting image, labeling and removing particles were also applied (Abràmoff et al. 2004).

Thereafter, the fiber objects and air bubbles were verified from the threshold image by using a 3D image visualization software (Bruker-micro CT) (Bruker-microCT 2013). However, in reality, some air bubbles might be attached to the fiber surfaces in the concrete. Image processing might not be able to separate these air bubbles, which might affect the results when analyzing the spatial distance between fibers. As a result, these air bubbles were manually removed. In addition, some large, non-spherical objects were visually interpreted as air bubbles by comparing with the original images. These manual adjustments were commonly applied in CT reconstruction to provide accurate image analysis (Bordelon and Roesler 2014).

4.4.3 Image analysis

A MATLAB program (Mathworks 2018) was developed to calculate the distance among the fiber clusters. When fibers were connected, they were identified as one fiber cluster. The connectivity is defined as the percentage of connecting fibers to the total fibers. However, as the program identified the fiber cluster by sphericity, Feret shape and volume of the object, it was necessary to manually check the accuracy before proceeding to the next step. Afterwards, the program would calculate the distance of each fiber to all other fibers or fiber clusters. Finally, the minimum distance from each fiber to all other fibers or fiber clusters could be obtained.

Connectivity obtained from different number of slices is illustrated in Fig. 4.3. It was found that when the number of slices was larger than 1700, the results were relatively stable in all the samples. In this study, it was found that using at least 2000 slices could provide consistent results.

Fig. 4.3. Connectivity in different UHPC samples.

To prevent spalling, vapor in the concrete should be released from concrete interior to exterior. This means that the interconnecting network formed by microcracks or fibers should percolate through the sample. By definition, when a fiber cluster percolated from the 1st slice to the 2000th slice (top surface to bottom surface), it was considered as percolation (Gu et al. 2016).

4.5 Result and discussion

4.5.1 Spalling test and permeability measurement

Spalling test results of UHPC with different fiber geometries and fiber dosages are presented in Fig. 4.4. As expected, UHPC-C samples spalled into very small pieces. With 3 kg/m³ of PP fibers, only U33/12/03 samples did not spall, while the other samples fragmented into large pieces, as shown in Fig. 4.4c to g. In addition, during the spalling test, UHPC-C samples spalled with a series of popping sounds, which lasted for more than ten minutes, while spalling happened with one or several sudden loud bangs in the spalled samples with fibers. The above findings affirmed that fiber geometry could affect the spalling resistance of UHPC significantly. Permeability

results (Fig. 4.5) revealed that permeability of U33/12/03 was much greater than that of UHPC-C at 150 °C and 200 °C, but the permeability values of U33/06/03, U100/06/03, and U100/12/03 were almost similar to that of UHPC-C. Although the number density of PP fibers in U33/06/03 was double compared to U33/12/03, the former did not show higher permeability values.

For PP fibers with a dimension of $\varnothing 100 \mu\text{m} \times 12 \text{ mm}$, the effectiveness of preventing spalling improved with increasing fiber dosage (Fig. 4.4e to h). When fiber content was low (below 6 kg/m^3), severe spalling occurred. With 9 kg/m^3 of PP fibers, spalling was mitigated somewhat, and most parts of the samples remained after the spalling test. A dosage of 12 kg/m^3 of PP fiber could prevent spalling of UHPC. As shown in Fig. 4.5, increasing fiber dosage could increase permeability of UHPC at 150 °C and 200 °C.

Fig. 4.4. Spalling test results: (a) UHPC-C, (b) U33/12/03, (c) U33/06/03, (d) U100/06/03, (e) U100/12/03, (f) U100/12/06, (g) U100/12/09, and (h) U100/12/12.

Fig. 4.5. Permeability of UHPC samples.

4.5.2 Fiber distribution

From X-ray CT results, it can be observed that PP fibers distributed randomly in samples with numerous air bubbles. Fig. 4.6a shows an example of fiber and air bubble distribution in a UHPC sample. PP fibers were in red, while air bubbles (which looked like small balls) were in blue. A small part of Fig. 4.6a was amplified in Fig. 4.6b, in which details of air bubbles and fibers could be clearly observed. As can be seen, most of the air bubbles were isolated, while some air bubbles were attached to fibers.

By using the image processing method mentioned in Section 4.4.2, air bubbles and PP fibers could be differentiated. Fig. 4.7 shows the fiber distribution in concrete samples. Fig. 4.7c and d clearly show that with 3 kg/m^3 of $100 \mu\text{m}$ diameter PP fibers, the number of fibers was relatively low. Fibers in U100/06/03 and U100/12/03 were scattered individually rather than connected as fiber clusters. Besides, it was also found that fibers were not dispersed uniformly. There were some regions without any fibers in U100/06/03 and U100/12/03. When the number of fibers increased, fibers

were easier to connect with one another and fiber networks could cover more regions in the UHPC samples. However, a high number density of fibers might cause balling or agglomeration of fibers.

Fig. 4.6. Fiber and air bubble distribution in U33/12/03 sample: (a) overview and (b) detail.

Fig. 4.7. Fiber distribution in different UHPC samples: (a) U33/12/03, (b) U33/06/03, (b) U100/06/03, (d) U100/12/03, (e) U100/12/06, (f) U100/12/09, and (g) U100/12/12.

As discussed in CHAPTER 3, microcracks caused by the expansion of fibers before melting is the key of increasing permeability of UHPC at high temperature. Microcracks are likely to connect to one another when fibers are close by, as shown in Fig. 4.8. In addition, as widely reported in the literature, when two fibers connect, the microcracks formed by these two fibers are likely to connect (Li et al. 2019). Thus, the spatial distribution of fibers (connectivity and distance) is curial for increasing permeability, as it may affect the connection of microcracks. The connectivity of fibers in different samples is summarized in Table 4.2. Generally, fibers with a high aspect ratio were more likely to connect and form fiber clusters. With a same dosage, the connectivity reduced significantly when fiber length reduced. Meanwhile, increasing fiber diameter resulted in a significant decrease in connectivity. In addition, it was found that increasing fiber dosage could increase the connectivity of fibers. In UHPC with fibers of 100 μm in diameter and 12 mm in length, when fiber dosage increased from 3 kg/m^3 to 12 kg/m^3 , connectivity of fibers increased from around 54.5 to 78.8 % (Table 4.2). If considering the number of connecting fibers, the increase was more significant.

Fig. 4.8. Typical connection of two microcracks.

Table 4.2. Connectivity and percolation of fiber clusters.

Samples	Connectivity (%)	Percolation cluster (%)
U33/06/03	43.9	-
U33/12/03	76.7	65.7
U100/06/03	18.6	-
U100/12/03	54.5	-
U100/12/06	54.6	-
U100/12/09	67.0	47.4
U100/12/12	78.8	57.3

Distances between fibers in the UHPC samples are shown in Fig. 4.9 and Fig. 4.10. As shown in Fig. 4.9a and Fig. 4.10a, with increasing the aspect ratio of fibers, distances between the fibers are reduced significantly. Besides, increasing fiber length could reduce the distance between fiber clusters. In U33/12/03, around 85% of fiber clusters were spaced less than 50 μm . But in U33/06/03, the percentage reduced considerably to around 68 %. Similarly, in U100/12/03, most of fiber clusters are spaced around 200 μm , while in U100/06/03, the majority of them are more than 200 μm away. In contrast, decreasing fiber diameter caused a significant reduction in inter-fiber distance. The spacing of fiber clusters in U33/12/03 and U33/06/03 is much smaller than that in U100/12/03 and U100/06/03, respectively.

Evidently, with a same fiber geometry, increasing fiber dosage also reduced fiber distance, as shown in Fig. 4.9b and Fig. 4.10b. However, fiber distance only reduced slightly when fiber dosage increased from 3 to 6 kg/m^3 . In U100/12/06, more fiber clusters were within 200 μm to other fiber clusters compared with that in U100/12/03. In addition, it was found that fiber connectivity did not show any change when fiber dosage increased from 3 to 6 kg/m^3 . However, it should be noted that as the number density of fibers is doubled, the number of fibers forming interconnecting network in U100/12/06 is much more than that in U100/12/03. Fiber connectivity increased significantly when fiber dosage increased from 6 to 9 kg/m^3 and from 9 to 12 kg/m^3 (Table 4.2), while the distance among fibers reduced (Fig. 4.10b). As evident, high

fiber connectivity and small fiber distance were beneficial for forming interconnecting network of microcracks. Therefore, U33/12/03, U100/12/09 and U100/12/12 showed higher permeability values than other samples.

Fig. 4.9. Cumulative percentage as a function of fiber distance in UHPC (a) with different fiber geometries and (b) different fiber dosages.

Fig. 4.10. Distance distribution of fiber clusters of UHPC with (a) different fiber geometries and (b) fiber dosages.

Fig. 4.11 shows the percolating fiber clusters in UHPC samples. It was found that percolation only existed in U33/12/03, U100/12/09, and U100/12/12. Obviously, high aspect ratio of fibers and high fiber dosages increased the connectivity and reduced the fiber distance, leading to the percolation of the system. With the percolation in the system, permeability of U33/12/03, U100/12/09, and U100/12/12 showed a

significant increase, thereby enhancing the spalling resistance (Fig. 4.4 and Fig. 4.5). It should be noted that the significant increase in permeability in samples of U33/12/03, U100/12/09, and U100/12/12 occurred at 150 °C. At this stage, microcracks were formed due to thermal mismatch between the fibers and then cement matrix. The distribution of microcracks was highly dependent on the distribution of fibers. Since the microcracks formed by two connected fibers are likely to connect (Li et al. 2019), percolation of fibers may result in percolation of microcracks, leading to an increase in permeability in these samples at 150 °C. Meanwhile, the number of percolating fibers could affect the number of percolating microcracks. As shown in Table 4.2, permeability in samples of U33/12/03, U100/12/09, and U100/12/12 increased with increasing proportion of percolating fiber cluster. The percentage of percolating fiber cluster was much higher in U100/12/12 than that in U100/12/09. Considering the lower number density of fibers in U100/12/09 than in U100/12/12, the number of fibers that were connected to percolating fiber cluster was even fewer. The microcracks associated with percolating fibers might not provide enough pathways in samples. As a result, permeability of U100/12/09 was much lower than that of U100/12/12 (Fig. 4.5). In addition, it was also found that the percolating cluster was more uniformly distributed in U100/12/12 and U33/12/03, rather than in U100/12/09. In U100/12/09, some regions were not covered by percolating fiber cluster. There were many isolated fibers or isolated fiber clusters. The fibers and their associated microcracks were more likely to be reservoirs rather than pathways for vapor. Vapor accumulated in these regions could not be released to the exterior, leading to partial spalling (Fig. 4.4g).

It was also found that permeability of U100/12/06 showed a significant increase, though there was no percolation. This might be caused by the connection of microcracks. As shown in Fig. 4.9 and Fig. 4.10, around 78 % of fibers in U100/12/06 were within a distance of 150 μm . Due to the small distance, microcracks generated

by fibers may connect and lead to percolation. This emphasizes the importance of microcracks and fiber spacings in percolation. However, it should be noted that spalling was not eliminated in this case due to the low permeability.

Fig. 4.11. Percolation of fiber cluster in UHPC samples: (a) U33/12/03, (b) U100/12/09, and (c) U100/12/12. Percolating fiber cluster is in green and isolated fiber cluster is in red.

For other samples (U33/06/03, U100/06/03, and U100/12/03), although many fibers could form fiber clusters, fibers, and their surrounding microcracks could not achieve percolation, due to low fiber connectivity and large distance among fibers. PP fibers and their surrounding microcracks in these samples acted as reservoirs rather than pathways for trapped vapor and had limited contribution to permeability. Therefore, permeability values of these samples did not show an obvious increase compared with UHPC-C. During heating, vapor in concrete may accumulate at some locations and caused spalling with a loud sound instead of simply popping. Hence, the remaining samples after spalling test were relatively larger in size than those in UHPC-C (Fig. 4.4).

4.5.3 Air bubble content and mechanical properties

An example of air bubble distribution is displayed in Fig. 4.12. Air bubble contents of UHPC samples are presented in Fig. 4.13. Generally, air bubble content showed the opposite trend with flowability (Fig. 4.14): the higher the flowability, the lower the air bubble content in the UHPC was. Lower flowability caused greater air entrainment during mixing. In U100/06/03 and U100/12/03, as the number density of fibers was relatively low, adding fibers did not affect flow and air bubble content of UHPC. Reducing fiber diameter or increasing fiber dosage, which increased number density of fibers, reduced the flow of sample, thus, leading to a significant increase in air bubble content.

Fig. 4.12. Air bubbles in U33/12/03 sample.

In addition, it was found that adding 3 kg/m^3 of PP fibers did not affect both 7-day and 28-day compressive strength significantly, as shown in Fig. 4.15. However, high fiber dosage, for example, 12 kg/m^3 of PP fibers ($100 \text{ }\mu\text{m}$ in diameter and 12 mm in length), reduced compressive strength by 11.6% and 16.9% in 7 and 28 days,

respectively, compared to UHPC-C. This is possibly due to the high air bubble content in the samples (He et al. 2017, Weng et al. 2018). Therefore, although high fiber dosage could prevent spalling of UHPC, the negative effects on flow and compressive strength should be considered.

In some studies, the extra air bubbles created by adding fibers were considered as one mechanism of PP fiber in preventing spalling (Khoury 2008). However, in this study, although U33/06/03 and U100/12/09 had high air bubble contents (Fig. 4.13), the samples spalled after exposure to ISO834 (Fig. 4.4), while U33/12/03 with a similar air bubble content did not spall. This suggests that the changes in air bubble content caused by adding fibers do not affect the spalling resistance of UHPC. Fig. 4.6b showed that most of the air bubbles were isolated and might only act as reservoirs during heating. In real cases, air bubbles might help connect microcracks and therefore enhance the connectivity of crack networks. But the test results (Fig. 4.11 and Table 4.2) clearly showed that the percolation and connectivity in the system were dominated by the distribution of PP fibers.

Fig. 4.13. Air bubble content of UHPC samples.

Fig. 4.14. Flow of UHPC samples.

Fig. 4.15. Compressive strength of UHPC with different sizes and different dosages of fibers.

Nonetheless, microcracks were not captured by X-ray CT in this work, as the resolution used was 7.8 μm . An X-ray CT with a submicron resolution was necessary to further investigate the effect of microcracks on the connectivity. However, the compromise between high resolution and size of samples may lead to a very small sample with a size of less than 1 mm (Bossa et al. 2015). To obtain a representative

sample of this small size, other measures are required.

In short, for the first time, this work shows the spatial distribution of fibers and investigates the effect of fiber connectivity on spalling prevention. The results presented here clearly show how fiber geometry and dosage affect inter-fiber connectivity, fiber distance and percolation of fibers, which further affect permeability and spalling behaviors of UHPC. Two aspects should be considered in choosing fibers for preventing spalling of UHPC: (1) long fibers to increase the probability of formation of fiber clusters, and (2) fine fibers or high fiber dosage to ensure percolation and provide adequate number of fibers that can contribute to pathways for trapped vapor.

4.6 Summary

The work presented in this chapter illustrates how the spatial distribution of PP fibers (fiber connectivity and cluster spacing) affects the spalling resistance of UHPC. From the results, the following conclusions are drawn:

- (1) At 150 °C, the formation of interconnecting network of microcracks, which are created by thermal expansion of PP fibers, was related to fiber distribution. High connectivity and small distance of fibers were beneficial for the formation of interconnecting networks of microcracks, leading to good spalling resistance. Percolating fiber clusters could result in percolation of microcracks, leading to a significant increase in permeability at 150 °C. Thus, percolation of PP fibers is crucial to increasing permeability, thereby enhancing spalling resistance. Furthermore, the number of percolating microcracks is related to the number of percolating fibers. The microcracks associated with percolating of fibers should provide adequate pathways to

achieve high permeability. This means that there is a requirement on the number of fibers that can contribute to forming a percolating interconnecting network.

- (2) Fiber geometry and fiber dosage affect spalling resistance. Increasing fiber aspect ratio (increasing fiber length or reducing fiber diameter) and fiber dosage can increase fiber connectivity and reduce the distance among fibers, leading to the percolation of fiber/crack system. Therefore, long and fine fibers show better performance in preventing spalling of UHPC than short and thick fibers. A much higher dosage is needed for thick fibers to achieve spalling resistance for UHPC.
- (3) Adding long fibers and high fiber dosage reduces flowability and increases air bubble content. But the changes in air bubble content caused by fiber inclusion do not seem to affect spalling resistance. A very high fiber dosage (12 kg/m^3 in this study) also causes a significant decrease in compressive strength of the system.

CHAPTER 5 CRITICAL FIBER DOSAGE FOR SPALLING PREVENTION

5.1 Overview

Based on the findings in CHAPTERS 3 and 4, the mismatch in the relative expansion of fibers and concrete matrix at high temperatures creates microcracks around fibers and forms interconnected networks in the concrete. This results in an increase in permeability and improvement in spalling resistance of UHPC. Fiber geometry and dosage are other key parameters influencing the connectivity and distance among fibers, in turn determining the percolation of interconnecting networks.

Therefore, a three-dimensional continuum Monte-Carlo model based on the Kozeny-Carman equation was developed to obtain the connectivity and percolation threshold of fiber/crack system. Further, a semi-empirical equation was proposed to determine the critical geometry and dosage of PP fibers required for preventing spalling of UHPC. The proposed approach has been verified by permeability and spalling tests.

5.2 Theoretical model based on Kozeny-Carman equation

The Kozeny-Carman equation is also widely used in petrophysics to relate permeability and porosity through different types of rocks and sandstones (Kozeny 1927, Carman 1939, Henderson et al. 2010, Latief and Fauzi 2012, Tiab and Donaldson 2015). This equation is expressed as:

$$k = \frac{\phi_p^3}{B\tau^2 S^2} \quad (5-1)$$

S is the surface area per unit volume (m^{-1}), and τ is the tortuosity of the medium. For

cracks in concrete, τ is estimated to be ~ 1.22 (according to (Akhavan et al. 2012)). The parameter B is the geometric factor (or Kozeny constant), which depends on the geometry of porous media. For circular tubes, B is usually taken as 2, while for cracks, $B=3$ (Ma 2015). In our current work, a value of $B=3$ is chosen to model the percolation of cracks resulted from the thermal mismatch of fibers and matrix at a temperature of 150 °C. As shown in our previous work in CHAPTER 3, permeability value at 150 °C is critical to preventing spalling.

The relative porosity of concrete is given by

$$\phi_p = \frac{V_p}{V_b} \quad (5-2)$$

where V_b and V_p are the bulk volume of concrete and pore volume in the concrete, respectively. In the proposed model, only the microcracks in concrete are considered. Since the crack tip is usually small, the cross section of microcrack is simplified to be a rectangle, as shown in Fig. 5.1a, so that the pore volume V_p is determined by the volume of microcracks V_c :

$$V_p = V_c = n_c w_c h_c l_c \quad (5-3)$$

where V_c is the volume of microcracks, n_c is the number of microcracks, w_c is the width of microcracks, h_c is the height of microcracks, and l_c is the length of the microcracks, as shown in Fig. 5.1b. It should be noted that at this temperature, the microcracks created by aggregates, if any, are small in number, and therefore, are neglected in the permeability calculations.

Though the thermal mismatch is all along the fibers resulting in the propagation of radial cracks from the fibers, in this study, it is assumed that each fiber could form one microcrack that propagates (or coalesce) along the whole fiber length ($l_f = l_c$,

Fig. 5.1b). This assumption is recognized in the literature as in (Delale 1988, Delale and Boduroglu 1991, Ho and Suo 1992). Fig. 5.2, a representative SEM micrograph, shows the presence of a microcrack along the fiber length. Nonetheless, as a result of the assumption, the number of microcracks equals to the number of fibers, which could be calculated by

$$n_c = n_f = \frac{V_b C_f}{V_f} \quad (5-4)$$

where n_c and n_f are respectively the number of microcracks and number of fibers, C_f is the volume percentage of PP fibers and V_f is the volume of individual PP fibers, which could be determined by the following equation:

$$V_f = \frac{\pi d_f^2}{4} \cdot l_f \quad (5-5)$$

where d_f is fiber diameter and l_f is fiber length.

Fig. 5.1. Sketch of microcrack in concrete: (a) cross-section and (b) microcrack along fiber.

Fig. 5.2. SEM micrograph on microcrack along fiber channel (After exposure to 150 °C, UHPC samples were cut and polished. The fiber remained in the channel was removed).

Substituting Eqs. (5-3), (5-4) and (5-5) into Eq. (5-2), the porosity of microcracks induced by expansion of PP fibers can be calculated by:

$$\phi_p = \frac{4w_c h_c C_f}{\pi d_f^2} \quad (5-6)$$

Assuming that the microcracks provide all of the permeability, the specific surface area per unit bulk volume is:

$$S = \frac{2n_c(w_c + h_c)l_c}{V_b} \quad (5-7)$$

Since microcrack width w_c is much smaller than microcrack height h_c , Eq. (5-7) is simplified to:

$$S \approx \frac{2n_c h_c l_c}{V_b} \quad (5-8)$$

Combining Eqs. (5-2), (5-3) and (5-8) yields:

$$S = \frac{2\phi_p}{w_c} \quad (5-9)$$

Substituting Eq. (5-9) into Eq. (5-1), it can be obtained that the permeability of concrete is

$$k = \frac{\phi_p w_c^2}{12\tau^2} \quad (5-10)$$

As the porous media is impermeable if the porosity is below the percolation threshold (Mavko and Nur 1997), this approach could only be used for the case that the porosity exceeds the percolation threshold.

In addition, it should be noted that isolated microcracks in concrete only serve as ‘reservoirs’ for trapped vapor. Some microcracks might connect and form an interconnecting cluster. Moreover, as long as the clusters do not percolate, they can only act as reservoirs instead. In this study, the crack clusters that percolate the system are defined as percolating cracks. As microcracks were related to fibers in this study, the number of percolating cracks were assumed to be equal to that of percolating fibers. For a sample with a fiber dosage of C_f , only a portion of it (C_{pf}), which represents the content of percolating fibers, contributes to increasing permeability. As a result, the term C_f in Eq. (5-6) is replaced by C_{pf} . Then, Eq. (5-6) is written as,

$$\phi_p = \frac{4w_c h_c C_{pf}}{\pi d_f^2} \quad (5-11)$$

From Eq. (5-11), microcrack width (w_c), microcrack height (h_c) and content of percolating fiber (C_{pf}) have to be determined.

In this study, microcrack width (w_c) is determined by the crack-tip opening displacement of cement paste. It is commonly accepted that w_c is a material constant dependent on the properties of concrete (Bažant and Oh 1983). According to Hillerborg et al. (1976), w_c is given by

$$w_c = \frac{2K_I^2}{E_c f_t} \quad (5-12)$$

where, K_I is the stress intensity factor of cement matrix, E_c is elastic modulus of

concrete and f_t is tensile strength. These parameters can be obtained from test measurements.

Based on the configuration of microcracks shown in Fig. 5.1a, the stress fields inducing radial cracks in matrix can be determined by (Delale 1988, Lu et al. 1991)

$$\sigma_{\theta\theta}^m = \sigma \left(\frac{R}{R + h_c} \right)^2 \quad (5-13)$$

where R is the radius of embedded fibers.

In this study, it is assumed that radials cracks stem from thermal mismatch. The stress can be calculated by (Lu et al. 1991)

$$\sigma = \varepsilon_T / \gamma \quad (5-14)$$

where ε_T is the thermal mismatch strain given by

$$\varepsilon_T = \int_{T_0}^T \Delta\alpha dT \quad (5-15)$$

where $\Delta\alpha$ is the difference in CTE between the PP fibers and the cement matrix. If the CTE of cement matrix is α_c and CTE of PP fiber is α_f , then $\Delta\alpha$ is given by $\Delta\alpha = \alpha_f - \alpha_c$.

Parameter γ from Eq. (5-14) can be calculated by (Delale 1988)

$$\gamma = \frac{1 + v_m}{E_c} + \frac{1 - v_f - 2v_f^2}{E_f} \quad (5-16)$$

where E_f is elastic modulus of embedded fiber at high temperature, v_m is Poisson's ratio of cement matrix which is 0.25 (Nguyen et al. 2019), and v_f is Poisson's ratio of PP fibers which is 0.42 (Wypych 2016).

Assuming there are no pre-existing cracks, a stress criterion was used in this study to determine the maximum crack height h_c (Leguillon 2002), which can be obtained by setting $\sigma_{\theta\theta}^m = f_t$.

However, it is difficult to determine the content of percolating fibers C_{pf} experimentally. In this study, a three-dimensional percolation model, which will be presented in Section 5.3, was developed to obtain C_{pf} .

5.3 Percolation model

To obtain percolation threshold and connectivity, a three-dimensional percolation model was developed using a cube constructed in Cartesian coordinate system. Microcracks were simplified as rectangular elements (Fig. 5.3). The elements simulating cracks were assumed to be straight with identical shapes in the percolation model (Li and Zhang 2009, Tran et al. 2013, Gu et al. 2016). It was also assumed that the elements were dispersed uniformly in the UHPC samples. Fig. 5.3 shows an example of the continuum model with 100 elements. Since the specimens used for permeability measurements had a thickness of around 50 mm, the length of the cube was set as 50 mm. The intersection of two elements was judged by using Geom3d library, which is a MATLAB toolbox developed by Legland (2018).

When the simulation started, the elements were generated randomly in the model one by one. The intersection of a newly generated element with the pre-existing elements was checked. In a local region, intersecting elements were labeled as one cluster. The new element was amalgamated into that cluster, if it intersected with any elements in the cluster. Elements that touched the upper or bottom boundary of the 50 mm cube were labeled as two special groups. When both the upper and the bottom boundary fibers were included in a cluster, the sample was assumed to be percolated

by this cluster of fibers (Gu et al. 2016). To save computational resources, the algorithm proposed by Li and Zhang (2009) was applied in the model and the algorithm proposed by Newman and Ziff (2001) was adopted to expedite the amalgamation of elements.

Fig. 5.3. Sketch of percolation model.

To determine the percolation threshold, the simulation was terminated when the system just attained percolation. The number of elements was recorded and the percolation threshold was defined as the minimum number of elements when percolation occurred for the very first time in the system (Gu et al. 2016). To ascertain the percolating fiber, the number of elements was pre-defined to be the number of fibers (determined by fiber dosage and geometries) before starting the numerical simulation. The simulation was terminated when the number of elements reached the pre-defined number. During the simulation, the elements that belonged to the percolating cluster was counted as N_{pf} . The fraction of percolating cracks p could be obtained by

$$p = \frac{N_{pf}}{N_f} \quad (5-17)$$

where N_f is the total number of fibers.

Similarly, when fiber dosage and fiber geometry was known, the number of elements could be obtained and p could also be defined as

$$p = \frac{C_{pf}}{C_f} \quad (5-18)$$

where C_f is the volume percentage of PP fibers and C_{pf} is content of percolating fiber.

An example of results on the fraction of percolation fibers is shown in Fig. 5.4, in which the program was implemented 100 times. It was found that only around 65.2 % of cracks (fibers) were percolating the cube in this example. The results were random and showed a distribution close to the normal distribution, with a mean value of 0.652 and a standard deviation of 8.42×10^{-3} , as shown in Fig. 5.5. As the standard deviation was relatively low, when using the model in Section 5.2 to predict permeability, the average value of p calculated by using percolation model will be adopted.

Fig. 5.4. An example of results of 100 implementations (element size: $l_f = 12$ mm, $h_c = 185 \mu\text{m}$, total number of fibers = 1.313×10^4).

Fig. 5.5. Histogram of fraction of percolating fibers for 100-time calculation (element size: $l_f = 12$ mm, $h_c = 185$ μm , total number of fibers = 1.313×10^4).

5.4 Experimental study

5.4.1 Material and specimen preparation

Material preparation mix proportion and raw materials used in this work were described in CHAPTER 3. In this current chapter, to understand the percolation threshold, PP fibers of different geometry were used: length variations of 6, 9, 12 and 18 mm; and diameter variations of 28 μm , 33 μm , and 100 μm . All the fibers had a density of 900 kg/m^3 . A dosage of 3 kg/m^3 (0.33 vol.%) was used for all these samples. Besides, as a representative composition, UHPC with PP fibers of 100 μm in diameter and 12 mm in length were cast with different fiber dosages of 0.66, 0.99 and 1.32 vol.%. UHPC without any fibers is named as UHPC-C. Other samples were named after the diameter, length and dosage of fibers, respectively. For example, U100/12/0.33 denoted UHPC samples with 100 μm in diameter, 12 mm in length and with 0.33 vol.% of PP fibers.

Gas permeability was measured on circular discs ($\varnothing 150$ mm $\times$ 50 mm) and spalling

tests were conducted on cylinders ($\varnothing 50 \text{ mm} \times 100 \text{ mm}$). The test procedures could be referred to CHAPTER 3.

5.4.2 Determination of parameters in the model

Some of the parameters that are required for the percolation model are determined experimentally. These include elastic modulus of cement matrix E_c (measured according to ASTM C469/C469M (ASTM 2014) by using specimens of $100 \text{ mm} \times 200 \text{ mm}$); tensile strength f_t (measured on samples with dimensions of $100 \text{ mm} \times 200 \text{ mm}$ according to ASTM C496/C496M (ASTM 2017)); and matrix intensity factor K_I (measured by three-point bending test following a procedure described in ASTM E399 (ASTM 2017)). This intensity factor is suitable for cementitious material and mortar with a maximum aggregate size of 1 mm (Ma et al. 2015). The dimensions of specimens for this test were $285 \text{ mm} \times 70 \text{ mm} \times 50 \text{ mm}$, with a notch-depth-to-height ratio of 0.4, as shown in Fig. 5.6. The test was conducted by using an MTS testing machine with a displacement control rate of 0.06 mm/min. The span between the two supports was maintained as 254 mm.

Thermal strain and respective CTE of PP fibers and cement paste and thermal strain were determined by thermomechanical analyzer, as described in CHAPTER 3. Dynamic mechanical analyzer (DMA, from TA Instruments, model: Q800) was adopted to determine the modulus of PP fibers (E_f) at 150 °C.

The properties of cement paste and PP fibers obtained from these tests were summarized in Table 5.1.

Fig. 5.6. The setup of three-point bending test and the specimen geometry.

Table 5.1. Properties of cement paste and PP fibers at 150 °C.

Samples	Properties	Test results
Concrete	E_c	34.8 ± 5.9 GPa
	f_t	6.8 ± 0.7 MPa
	K_I	0.5 ± 0.07 MPa·m ^{1/2}
	α_c	$-4.0 \times 10^{-6} / K$ (shrinkage)
PP fibers	E_f	267.5 ± 5.9 MPa
	α_f	$8.4 \times 10^{-4} / K$
	ε_f	0.10

5.5 Results and discussion

Fig. 5.7 shows the permeability values of UHPC samples at high temperature. For all the samples, the permeability increased with increasing temperature. In the UHPC with PP fibers, the permeability usually showed a significant increase at 150 °C, and

beyond 150 °C, the rate (gradient) reduced. The behavior of PP fibers in UHPC had been investigated in detail in CHAPTER 3 and will not be discussed further here. From the spalling test results in Table 5.2 and Fig. 5.7, the permeability values of non-spalled samples increased by around two orders of magnitude at 150 °C relative to that at 105 °C. In contrast, the increases of permeability in spalled samples were only one order of magnitude or less, similar to that of UHPC-C. From Fig. 5.7a and b, although the permeability values of some spalled samples (U33/06/0.33 and U28/06/0.33) increased significantly at 200 °C and 300 °C, it was clearly showed that the increase in permeability at 200 °C and 300 °C did not result in good spalling resistance. It reaffirms that permeability at 150 °C is critical to preventing spalling. In addition, it was also found that the permeability values at 150 °C increased significantly when fiber length increased from 6 mm to 9 mm, while the permeability values did not change much when fiber length increased from 12 mm to 18 mm, as shown in Fig. 5.7 a and b.

As defined in Section 5.3, percolation threshold of UHPC samples with PP fibers is taken as the minimum volume percentage of fibers required to percolate the entire sample. The percolation thresholds of samples with different aspect (l_f/d_f) ratio is plotted in Fig. 5.8. As expected, percolation threshold decreased with increasing l_f/d_f ratio. When $l_f/d_f > 600$, the effect of l_f/d_f ratio on percolation threshold became negligible, while when $l_f/d_f < 150$, percolation threshold increased dramatically.

Fig. 5.7. Permeability with increased temperature: (a) UHPC with 28 μm PP fibers, (b)UHPC with 33 μm PP fibers and (c)UHPC with 100 μm PP fibers.

Table 5.2. Results of spalling tests.

Length	Diameter		
	28 μm	33 μm	100 μm
6 mm
9 mm
12 mm
18 mm

Permeability of UHPC samples at 150 °C is also plotted in Fig. 5.8. As can be seen, when fiber dosage was lower than percolation threshold, permeability did not show a significant increase compared with that of UHPC-C. For example, a dosage of 0.33 vol.% of PP fibers with a diameter of 100 μm was well below the percolation threshold (Table 5.3). PP fibers did not contribute to an increase in permeability due to failure in percolating the sample. As a result, permeability of UHPC with 100 μm of PP fibers was similar to that of UHPC-C and all these samples spalled regardless of fiber length (Table 5.2). Similarly, in U33/06/0.33, 0.33 vol.% of PP fibers were also below the percolation threshold (Table 5.3), so that PP fibers did not help prevent spalling (Table 5.2). In contrast, when fiber dosage was higher than percolation

threshold, adding PP fiber could lead to a significant increase in permeability. This emphasizes the importance of percolation for enhancing permeability. Therefore, considering a fiber dosage higher than percolation threshold in the model mentioned in Section 2 is reasonable. However, it should be noted that a fiber dosage higher than percolation threshold could not guarantee a non-spalling behavior (for instance, U33/09/0.33 and U28/06/0.33 (Table 5.2) as well as U100/12/0.66 and U100/12/0.99 (spalling test results refer to Fig. 4.4f and g)). In fact, it is the fraction of percolating cracks p that will determine the effectiveness of spalling prevention. The critical fiber dosage for spalling prevention will be further discussed in this section.

Fig. 5.8. Percolation threshold of fibers with different aspect ratios (l_f/d_f).

Table 5.3. Percolation threshold of fibers used in this study.

Diameter (μm)	Length (mm)	Percolation threshold (vol.%)	Diameter (μm)	Length (mm)	Percolation threshold (vol.%)	Diameter (μm)	Length (mm)	Percolation threshold (vol.%)
28	6	0.32	33	6	0.39	100	6	1.32
28	9	0.21	33	9	0.25	100	9	0.87
28	12	0.17	33	12	0.18	100	12	0.64
28	18	0.1	33	18	0.12	100	18	0.47

Fig. 5.9. Comparison of predicted results and test results.

Fig. 5.9 shows the comparison between the test results and the model predictions on the permeability of UHPC at 150 °C. The variations in the test results, which were quite common in permeability test results of UHPC with fibers (Li et al. 2019), might be caused by the inhomogeneity of concrete samples and nonuniform dispersion of fibers. Nonetheless, as shown in Fig. 5.9, the predicted values were comparable with the measured results and the trend of model predictions was similar to test results when fiber geometries changed.

The objective of adding PP fibers is to prevent spalling of UHPC at high temperature. Therefore, in actual applications, it is vital to choose fiber geometry and dosage to achieve good spalling resistance in UHPC. Theoretically, PP fibers with any geometry could prevent spalling of UHPC, as long as the fiber dosage is high enough. According to the literature (Klingsch et al. 2013, Zhang et al. 2018, Li et al. 2019) and test results in Section 5.5, it was found that when permeability is larger than $0.6 \times 10^{-16} \text{ m}^2$ at 150 °C, spalling could be eliminated. Therefore, a permeability value of $0.6 \times 10^{-16} \text{ m}^2$ was used as a reference. By adopting the model derived from Section

5.2, the percolating fibers (C_{pf}) for each geometry of fibers to achieve a permeability value of $0.6 \times 10^{-16} \text{ m}^2$ could be obtained. Subsequently, the percolation based non-spalling (PBN) fiber dosages for different geometries of fibers could be calculated using the proposed percolation model. The PBN fiber dosage was denoted as C_{PBN} , which was the requirement of non-spalling fiber dosage for different geometries of PP fibers.

Fig. 5.10. Relationship between C_{PBN} and l_f/d_f .

The relationship between C_{PBN} and the aspect ratio l_f/d_f is plotted in Fig. 5.10. However, it is worth noting that at high l_f/d_f ratio >600 , increasing l_f/d_f ratio did not result in much changes in C_{PBN} . In contrast, when the l_f/d_f ratio was lower than 150, reducing l_f/d_f ratio could cause a significant increase in C_{PBN} . It has been widely reported that high fiber dosage would reduce compressive strength and workability of concrete, and large fiber length may also reduce workability (Heo et al. 2010, Heo et al. 2012, Li et al. 2019). Besides, a parametric study also showed that increasing the fiber length by itself did not always result in an increase in permeability. In most cases, after fiber length was larger than 18 mm, further increasing the length did not improve permeability of UHPC, as shown in Fig. 5.11.

As a result, PP fibers with a l_f/d_f ratio between 300 to 600 would be favorable for preventing spalling of UHPC. It was also found that for the same l_f/d_f ratio, short and fine fibers could reduce C_{PBN} slightly compared with thick and long fibers. This could be due to the significant increase in number of fibers when reducing diameter and length.

Fig. 5.11. Predicted permeability with different fiber geometries.

A least-squares fitting curve is also plotted in Fig. 5.10. This fitting curve can be expressed by

$$C_{PBN} = 55.12(l_f/d_f)^{-0.87} \quad (5-19)$$

To validate the accuracy of Eq.(5-19), fibers with different geometries and different dosages were cast in UHPC to make cylinder samples and spalling tests were conducted in accordance with the same procedure mentioned in Section 5.4.1. Fig. 5.12 plots the test results conducted in this study as well as some spalling test results reported in the literature (Heinz et al. 2004, Klingsch et al. 2013, Ozawa et al. 2014, Sun and Lin 2016, Liang et al. 2018, Li et al. 2019, Missemer et al. 2019, Ozawa et al. 2019). In general, Eq. (5-19) could predict non-spalling fiber dosage for PP fibers

with different geometries with acceptable accuracy. Meanwhile, it is worthy to note that although Eq. (5-19) was based on the material properties used in this study, it could also give good predictions for the spalling test results conducted by other researchers on UHPCs with other mix designs. However, spalling test results could also be affected by many factors, e.g. heating rate, specimen dimensions, size and type of aggregates, dispersion of fibers, steel fibers, initial moisture content, etc. The influence of these parameters will be investigated in future work. Further, there are also some drawbacks in the model proposed in Section 5.2. For instance, some parameters, like multi-radial cracks, cracks caused by aggregates, and connectivity of cracks by air bubbles were ignored in the model. These will make the predicted results too conservative. Nevertheless, the approach proposed by this work provides meaningful insights and a simple methodology to estimate the efficacy of required fiber dosage for PP fibers with different geometries to prevent spalling. By using Eq. (5-19), it is possible to avoid choosing PP fibers that require very high fiber dosages to prevent spalling.

Fig. 5.12. Comparison of the spalling test results and C_{PBN} curve (green symbol: no spalling, red symbol: spalling).

5.6 Summary

Optimization of fiber geometry and dosage for spalling prevention of UHPC was conducted in this Chapter. Permeability measurements of UHPC with different fiber geometries and dosages were evaluated through tests. A percolation model was proposed to estimate the percolation threshold and connectivity of fibers/cracks in UHPC. Results showed that a PP fiber dosage lower than percolation threshold had limited effect on improving permeability and spalling resistance of UHPC at high temperatures.

An approach based on the Kozney-Carmon equation was developed to predict the permeability of UHPC with PP fibers of a dosage larger than the percolation threshold. The approach was calibrated with experimental data.

Other contributions of this Chapter include the proposition of a semi-empirical equation (Eq.(5-19)) to estimate PBN fiber dosages for preventing spalling of UHPC with different fiber geometries. In addition, it was also found that the PBN fiber dosage for preventing spalling remained more or less constant when l_f/d_f ratio of PP fiber was larger than 600, but it increased significantly with decreasing l_f/d_f ratio when l_f/d_f ratio of PP fiber was lower than 150. Based on this work, an aspect ratio between 300 and 600 was recommended for spalling prevention of UHPC. Although the parameters used in the current study might be specific to a certain range of UHPCs and PP fibers, the semi-empirical equation for PBN fiber dosage was validated by a series of spalling test results conducted at NTU and reported tests gleaned from the literature by other researchers. This was an improvement to the current guidelines in Eurocode 2, in which only a value of 2 kg/m³ PP fibers was recommended regardless of the fiber geometry. As illustrated in CHAPTER 2, it was reported that adopting this dosage could not prevent spalling in many cases.

CHAPTER 6 EFFECTS OF JUTE FIBERS ON FIRE RESISTANCE OF UHPC

6.1 Overview

From CHAPTERS 3 to 5, it has been established that the addition of synthetic fibers like PP enhanced the spalling resistance of UHPC. There is a growing emphasis on the usage of sustainable materials in the construction industry. From this viewpoint, replacing synthetic fibers with natural fibers like hemp, palm, flax, and jute would be an excellent step forward (Silva et al. 2011, Wei and Meyer 2015). So far, only some preliminary studies have been reported on the usage of natural fibers in UHPC (Ozawa et al. 2013, Ozawa et al. 2019).

This chapter focuses on the performance of UHPC with different dosages of jute fibers at elevated temperatures. The performance is evaluated in terms of residual mechanical properties, hot permeability, spalling resistance, and by exposed to accelerated weathering conditions. The investigation of these different properties/aspects under various exposure conditions is crucial for validating the usage of natural fibers in UHPC.

6.2 Design of experiments

6.2.1 Mix proportion and sample preparation

Mix proportion, sample preparation and raw materials of UHPC could be referred to CHAPTER 3. Different jute fiber dosages of 3, 5, and 10 kg/m³ were used in this study. The mix proportion of UHPC with different jute fiber dosages is shown in Table 6.1. Jute fibers were first soaked in 5 % NaOH solution for 24 h and then

washed with tap water until the pH of water passing through the fibers was ~ 7 . Subsequently, the fibers were dried in an oven at 60°C for 6 h. This pretreatment was aimed to remove the impurities and oil residues (Roy et al. 2012, Yan et al. 2012). After pretreatment, jute fibers were cut to ~ 12 mm in length. The diameter of fibers was in the range of 40 to $80\ \mu\text{m}$.

Table 6.1. Mix proportions of UHPC with different dosages of jute fibers.

Mix	W/B	C	SF	SS	SP	FA	Jute fibers (kg/m^3)
UHPC-C	0.2	1	0.25	0.25	0.06	1.1	0
UHPC-J3	0.2	1	0.25	0.25	0.06	1.1	3
UHPC-J5	0.2	1	0.25	0.25	0.06	1.1	5
UHPC-J10	0.2	1	0.25	0.25	0.06	1.1	10

*W/B: water to binder ratio, C: cement, AG: aggregates, SS: silica sand, SF: silica fume, SP: superplasticizer. UHPC-C denotes the control sample without jute fibers; other samples were named based on jute fiber content. For example, UHPC-J3 contains $3\ \text{kg}/\text{m}^3$ of jute fibers.

Fig. 6.1. Jute fibers used in this study.

Fig. 6.2. SEM micrographs of the jute fiber: (a) surface and (b) cross-section.

6.2.2 Compressive strength and mass loss

Compressive strength tests were conducted on 50 mm cubic specimens exposed to high temperatures varying from room temperature to 200, 400, 600, and 800 °C. The specimens were heated to target temperature using an electric furnace with a heating rate of 0.5 °C/min and then maintained at that temperature for 2 h. Previous studies have shown that with this heating rate and conditioning time, samples could reach an isothermal state (Liu et al. 2017). To minimize spalling, specimens exposed to 400, 600, and 800 °C were maintained at 200 °C for 2 h before being heated to the target temperature. Subsequently, the specimens were allowed to cool down to room temperature naturally in the furnace. Compressive strength tests at room temperature were performed at 28 days and the heating procedure also started at 28 days. The residual compressive strength was evaluated by using a hydraulic press machine with a loading rate of 100 kN/min. The peak loads were recorded, and compressive strength was reported as the mean value of three specimens for each target temperature. In addition, mass differences of samples before and after heating were recorded.

6.2.3 Flexural strength

Flexural strength was evaluated using 40 mm × 40 mm × 160 mm prisms with a clear span of 120 mm. The specimens were initially subjected to 200, 400, and 600 °C. Specimens were heated followed the same procedure in Section 6.2.2. The tests were conducted by using an MTS testing machine with a displacement-controlled rate of 0.25 mm/min. A 50 N preloading was applied to close any potential gaps in the loading apparatus. Average values of three specimens were reported for flexural strength.

6.2.4 Gas permeability test

The samples with a thickness of 50 mm and a diameter of 150 mm were used for permeability test. Permeability of samples at 105, 150, 200, and 300 °C was measured at hot state. The test procedure was introduced in Section 3.2.5. For each mix, at least two specimens were tested.

6.2.5 Spalling tests

Spalling tests were conducted on cylinders with 50 mm diameter and 100 mm height. Three cylinders were tested for each mix design (Table 6.1). The heating curve and test procedure were in accordance with Section 3.2.6.

6.2.6 Accelerated weathering test

To investigate the durability of UHPC with jute fibers and fiber degradation in UHPC matrix, UHPC-J10 samples were exposed to two different aggressive environments: 30 soak-dry cycles and 60 UV-spray cycles. Samples exposed to soak-dry cycling were denoted as UHPC-J10-SD. One soak-dry cycle includes 18 h of immersion in tap water at ambient temperature and followed by 6 h drying in an oven at 60 °C (British Standards Institution 2012). UV-spray cycling treatment was conducted in a QUV accelerated weathering tester (from Q-Lab) and the samples were denoted as UHPC-J10-QUV. Each UV-spray cycle comprises an 8h UV exposure at 60 °C, a 0.25 h water spray and 3.75 h of condensation (ASTM 2016). The irradiance of UV was set as 1.55 W/m² to simulate tropical conditions. Accelerated weathering tests were started immediately after the 28-day water curing after casting. Another group of samples was stored in the laboratory environment as a reference group and denoted as UHPC-J10-R. Compressive strength evaluation and spalling tests were carried out

on these weathered samples to understand the effects of different exposure conditions.

6.2.7 Thermal analysis

Differential scanning calorimeter (DSC) and thermogravimetric analysis (TGA) were conducted on jute fibers by using simultaneous thermal analyzer (SDT, from TA Instruments, model: Q600). A constant heating rate of 5 °C/min from room temperature to 900 °C was employed in DSC and TGA in either nitrogen or air atmosphere with a gas flow of 100ml/min.

Thermomechanical analyzer (TMA, from TA Instruments, model: Q400) was employed to investigate dimensional changes of jute fibers at high temperature. One jute fiber (of dimensions 12 mm by 61.3 µm) was placed between two quartz planes and put into the furnace. The furnace was heated to 300 °C with a heating rate of 5 °C/min under nitrogen atmosphere.

In addition, TGA and DSC tests were also conducted on UHPC with different fiber contents at a heating rate of 5 °C/min from room temperature to 900 °C in the air atmosphere with a gas flow of 100 ml/min.

6.2.8 SEM and X-ray computed tomography (X-ray CT)

SEM and X-ray CT observation were conducted on UHPC samples. The procedures could be referred to Section 3.2.4 and Section 4.4.1.

6.3 Results and discussion

To investigate the effects of incorporating natural fibers in UHPC on its explosive spalling behavior, it is important to understand the changes in (a) mechanical

properties of UHPC in the presence of different dosages of fibers, (b) fiber dimensionality with temperature, (c) percolation of fibers in the matrix with different fiber dosage, and (d) changes in water vapor permeability of the system with temperature. The following sections will focus on elaborating these changes while correlating the behavior with more conventional UHPC with PP fibers. Further, preliminary efforts are made to derive knowledge on the effects of exposure to accelerated weathering conditions to illustrate the durability of the system and its behavior.

6.3.1 Mass loss

Fig. 6.3 shows the mass loss of UHPC with different jute fiber dosages as temperature rises. At 200 °C, the mass loss of UHPC increased with growing fiber dosage. The mass loss of UHPC-C, UHPC-J3, UHPC-J5 and UHPC-J10 were 3.34 %, 5.56 %, 6.31 % and 7.82 %, respectively. The mass loss at this stage was caused by the evaporation of free water, gel water of calcium silicate hydrate (C-S-H) and water in ettringite (Fares et al. 2010, Abid et al. 2017). Since UHPC is very dense, it was difficult for the moisture in the concrete to escape, resulting in a low mass loss in UHPC-C. It is very interesting to find that the difference in the mass loss among different samples only occurred at 200 °C. After exposed to 400 °C, the mass loss of all the samples achieved around 10 %. Due to thermal incompatibility among the components of UHPC and shrinkage of cement matrix, microcracks were formed during heating. Consequently, the effect of jute fibers on mass loss of UHPC became negligible. Fiber dosage did not affect mass loss after 400 °C. There was no obvious mass loss between 400 °C and 600 °C. After being exposed to 800 °C, another approximate 2 % of mass loss could be gained in all the samples.

Fig. 6.3. Mass loss after exposure to different temperatures.

6.3.2 Failure pattern after compressive test

Fig. 6.4 shows the failure pattern of UHPC-C and UHPC-J10 after compressive strength tests. UHPC-C showed a brittle failure both at ambient temperature and after exposure to high temperature (Fig. 6.4a). At ambient temperature and after exposure to 200 °C, UHPC with jute fibers remained intact after compression test. There were many cracks on the specimen surface after the test. Above 400 °C, UHPC with jute fibers crushed when tested (Fig. 6.4b).

Fig. 6.4. Failure pattern of (a) UHPC-C and (b) UHPC-J10 after compressive strength.

6.3.3 Compressive strength

Compressive strength of UHPC with different fiber dosages after exposure to different temperatures is given in Fig. 6.5. As evident, at ambient conditions, the addition of fibers had no noticeable influence on compressive strength, even with a fiber dosage of 3 kg/m^3 . With a further increase in fiber dosage, a reduction in strength is noted. Up to 13 % drop has been observed with a fiber dosage of 10 kg/m^3 . This is understandable, considering the poor workability with higher dosages of fibers, resulting in their agglomeration and poor distribution in UHPC. This increases the porosity in the system, negatively affecting the compressive strength. To add on, the drying shrinkage of the swollen jute fibers and cement matrix could also induce interfacial voids between them (Wei and Meyer 2015) (see Fig. 6.9a and associated discussions for more information).

After the samples were exposed to 200°C , the residual compressive strength values have increased irrespective of the presence or absence of fibers. The strength of UHPC-C, UHPC-J3, UHPC-J5, and UHPC-J10 has increased by 20.8 %, 20.6 %, 24.3 %, and 41.7 %, respectively, compared to their corresponding values at the ambient temperature of around 25°C . This can be attributed to the accelerated pozzolanic reaction and rehydration of residual and unhydrated cement at a higher temperature of 200°C . This is a well-reported phenomenon in the literature (Chen and Liu 2004, Zheng et al. 2014).

Fig. 6.5. Residual compressive strength of UHPC with jute fibers exposed to high temperature.

More interestingly, the strength values did not show any noticeable drop in UHPC-C and UHPC-J3 even after exposure to 400 °C. However, with further increase in fiber dosages (UHPC-J5 and UHPC-J10), the extent of drop in compressive strength has increased relative to 200 °C. UHPC-J5 has shown a reduction in strength by ~6.8 %, whereas UHPC-J10 by ~24 %. From TGA data on jute fibers, it showed a huge mass loss of ~64 % between 250-400 °C (Fig. 6.6a), which might have triggered the negative effect on compressive strength of the UHPC system. It is also expected that instead of oxidation, pyrolysis could be predominant in the oxygen-depleted atmosphere as the fibers are embedded inside the concrete. After the initial evaporation of physisorbed/bound water from natural fibers (50-150 °C), the mass loss between 250-400 °C is typically attributed to the decomposition of hemicellulose and α -cellulose (Ray et al. 2002, Wei and Meyer 2015). As evident, the TGA data of fibers in N₂ atmosphere, as well as the simultaneous TGA-DSC data of UHPC with and without different fiber dosages (Fig. 6.7b), confirm this behavior.

Fig. 6.6. TGA-DSC results of (a) jute fibers and (b) UHPC with different dosages of jute fiber.

After exposure to 600 °C and 800 °C, the compressive strength values of all systems have dropped significantly relative to their respective values at 200 °C or even 400 °C. UHPC-C still had the highest compressive strength (78.97 MPa), which is a drop of 41.3 % from 134.6 MPa at ambient temperature; while UHPC containing 10 kg/m³ of fibers shows a compressive strength of 51.77 MPa (a 56.4 % drop compared to its ambient strength). Nonetheless, the reduction in the strength is not a direct consequence of the presence of fibers, but rather a combination of the result of

degradation of UHPC and fibers. It is well known that thermo-chemical changes that occur in concrete (dehydration, phase changes, shrinkage, aggregate expansion) will influence the deterioration and crack development (Abid et al. 2017). Furthermore, C-S-H phase, which is responsible for the strength of cement, decomposes from above 600 °C. It has been reported that after exposure in 600 °C and 800°C for 2 h, the proportion of decomposed C-S-H accounted for around 20 % and nearly 100 %, respectively (Peng and Huang 2008). All these factors result in a significant decrease in mechanical properties of concrete at high temperatures (Peng and Huang 2008, Fares et al. 2010, Peng and Yang 2017).

6.3.4 Flexural strength

Residual flexural strength of UHPC with different dosages of jute fiber after being subjected to different temperatures is shown in Fig. 6.7. At ambient temperature, jute fibers did not affect flexural strength of UHPC significantly with a fiber content of 3 kg/m³. With the increase of fiber dosage, the flexural strength reduced. About 10 % of reduction could be observed when the fiber dosage was 10 kg/m³. The weak interface between jute fibers and cement matrix and high porosity caused by poor workability can result in the reduction of flexural strength on the UHPC with high jute fiber dosage. After being exposed to 200 °C, the flexural strength decreased in all samples. More interestingly, UHPC with jute fibers showed a lower reduction in flexural strength than UHPC-C. UHPC-J10 only showed around 6 % of reduction, while UHPC-C had 39 % of reduction in flexural strength. The higher the fiber dosage, the smaller was the reduction and the greater was the flexural strength. At 400 °C, flexural strength of UHPC with jute fibers also showed a low reducing rate, while flexural strength of UHPC-C kept decreasing sharply. It may be because natural fibers could mitigate the formation of microcracks in the concrete during heating, as reported by Netinger Grubeša et al. (2018). However, it was found that flexural

strength of UHPC with jute fibers decreased more significantly than that of UHPC-C at 600 °C. This is caused by the combination of degradation of jute fibers and formation of microcracks in UHPC. But the flexural strength of UHPC with jute fibers was all higher than that of UHPC-C at the temperature of 600 °C.

Fig. 6.7. Residual flexural strength of UHPC with jute fibers exposed to high temperature.

6.3.5 Spalling behavior

An important aspect of this work is to understand the influence of jute fibers on resisting the explosive spalling behavior of UHPC. As per the guidelines suggested in CHAPTERS 3 to 5, the requirements for the materials to be used as additives in UHPC to promote permeability at higher temperatures are: (i) exhibit a higher magnitude of thermal mismatch with the cement matrix, and (ii) high aspect ratio of the fibers as this influences the crack density and interconnectivity. With regard to the first parameter, in typical synthetic polymers, specific volume in the polymeric system increases as temperature increases. For example, in polypropylene, it is noted that the linear change can be as high as 30-45 times between temperatures of 120-170

°C. This results in thermal mismatch with cementitious matrix. However, in the case of natural fibers, it is known that fibers shrink as the water bounded by them is lost, resulting in the collapse of cell walls. This is evident in the TMA data shown in Fig. 6.8. The shrinkage was about 5.8 % at 200 °C. In fact, the water absorbed by the fibers during the initial mixing/casting processes might result in swelling of the fibers, and during the hydration, the enthalpy of hydration process could as well result in the deswelling of fibers. This swelling/deswelling could as well result in interfacial separation of the fibers from the matrix. SEM micrographs showing this behavior between cement matrix and natural fibers even at room temperature are presented in Fig. 6.9a. With temperature increasing, the shrinkage of fibers, as expected, increased (Fig. 6.9b). Furthermore, as the bonding between the matrix and fibers was only expected to be weak, the separation by itself did not induce any cracks in the system (Fig. 6.9b). In this case, only the space between cement matrix and jute fibers could be the pathway of vapor at high temperatures. Therefore, the effectiveness of natural fibers was a critical question, particularly at low fiber loading levels where percolation of fibers could not be expected (influencing the second parameter mentioned above). As a result of this, only the fiber content of 10 kg/m³ could prevent spalling of UHPC in this study (Fig. 6.10).

Fig. 6.8. TMA result of jute fiber and cement matrix.

Fig. 6.9. SEM micrograph of jute fiber in UHPC at (a) ambient temperature and (b) 200 °C.

Indeed, the permeability data of UHPC/natural fiber systems confirms the above discussions. With a fiber dosage of 10 kg/m^3 , the permeability of UHPC-J10 is significantly high (Fig. 6.11). Compared to UHPC-C, permeability of UHPC-J10 is more than 40 times at 105°C , with an absolute value of $18.6 \times 10^{-18} \text{ m}^2$. It increased further by an order of magnitude reaching a value of $38.2 \times 10^{-17} \text{ m}^2$ at 200°C . This is due to the percolation of fibers (resulting in a significant increase in permeability as they shrink with temperature). This differs from percolation of network cracks produced as a result of thermal mismatch in the case of synthetic fibers like PP fibers

mentioned in CHAPTER 3. In UHPC/PP system, with the dimensions of PP fibers similar to jute fibers used in this study, even at a fiber dosage level of 3 kg/m^3 , the permeability data at 200°C is around $30 \times 10^{-17} \text{ m}^2$ (Li et al. 2018). This value is similar to UHPC with 10 kg/m^3 of natural fibers at 200°C . Consequently, the spalling behavior of UHPC is only prevented at this fiber dosage (10 kg/m^3) as compared to the fiber dosages of 3 and 5 kg/m^3 (Fig. 6.10). UHPC-C, as expected, exhibited severe spalling with the entire specimen breaking into small pieces. Though with temperature cement paste shrinks due to the loss of water molecules and aggregates slightly expand (around 0.26 % at 200°C and 0.35 % at 300°C) (in CHAPTER 3) thermal mismatch between the cement paste and the aggregates is not enough to significantly increase the permeability (Fig. 6.11).

Fig. 6.10. Spalling test results of UHPC with different jute fiber dosages: (a) UHPC-C, (b) UHPC-J3, (c) UHPC-J5, and (d) UHPC-J10.

Fig. 6.11. Hot permeability of UHPC with different fiber dosages.

Fig. 6.12. Percolation of jute fibers in (a) UHPC-J5 and (b) UHPC-J10 (the volume of prism: 7 mm × 7 mm × 15.7 mm). The spheres in the figures are air bubbles trapped in the system.

Based on the 3D stick percolation model (Gu et al. 2016), according to our calculations for fibers with a diameter in the range of 40-80 μm and length of 12 mm, a number density of 0.085-0.1 /mm³ is required to percolate a cubic volume of space.

Considering this, the percolation threshold for jute fibers is around 3.5-5.7 kg/m³. Despite this, as shown in Fig. 6.12, due to the poor distribution of fibers, the interconnectivity of fibers (and therefore, percolation) is better with fiber dosage of 10 kg/m³ than 5 kg/m³. Therefore, the permeability of UHPC-J5, though higher than UHPC-C (Fig. 6.11), was still not enough to prevent spalling in the current study.

6.3.6 Effect of accelerated weathering curing

This section illustrates our preliminary efforts in understanding the effects of weathering conditions on the different aspects of durability of UHPC with jute fibers. For this, only a representative fiber dosage level of 10 kg/m³ is chosen as it is the only sample that resists spalling. As explained in the experimental section, a set of samples was exposed to 60 cycles of accelerated weathering conditions in a QUV chamber, another set was exposed to 30 cycles of water soaking-drying cycles, and another was set aside under laboratory conditions (25 °C, 60 % RH) for 30 days. XRD spectra of these different sets of samples are shown in Fig. 6.13. As evident, there are no noticeable differences in the spectra of different samples. This suggests that different accelerated weathering treatments did not cause any phase/chemical changes in UHPC with jute fibers. In fact, even microstructural changes could not be identified after exposure to weathering conditions, as shown in Fig. 6.14.

The compressive strength values of UHPC-J10 samples after different accelerated weathering are shown in Table 6.2. As can be seen, compressive strength of all the samples increased by up to 35 % compared to the value at 28 days. However, compressive strength of UHPC-J10-SD and UHPC-J10-QUV show a slight reduction of 8.7 % and 7.4 %, respectively, compared with UHPC-J10-R. This might be a result of microcracks that form during accelerated weathering cycles, in particular, with the temperature changes during each cycle. Spalling tests on these different sets of

samples further confirmed that the durability has not been compromised, as none of the samples spalled, as shown in Fig. 6.15.

Fig. 6.13. XRD results of UHPC-J10 samples after accelerating aging.

Fig. 6.14. SEM micrograph of jute fiber in UHPC after accelerated weathering curing: (a) UHPC-J10-SD and (b) UHPC-J10-QUV.

Table 6.2. Mechanical properties of UHPC after accelerated weathering curing.

Samples	Compressive strength (MPa)	Flexural strength (MPa)
UHPC-J10 (28 days)	118.3 ± 2.63	13.86 ± 0.47
UHPC-J10-R	160.50 ± 2.03	11.88 ± 0.28
UHPC-J10-SD	146.47 ± 7.01	12.63 ± 1.87
UHPC-J10-QUV	148.57 ± 11.65	12.61 ± 1.06

Fig. 6.15. Spalling test after accelerated weathering curing: (a) UHPC-J10-SD and (b) UHPC-J10-QUV.

6.4 Summary

This chapter investigated the effects of jute fiber content on residual mechanical properties and spalling resistance of UHPC. This resulted in the following conclusions:

- (1) As the natural fibers shrunk with temperature, the percolation of fibers in the matrix is critical to resisting thermal spalling in UHPC. It is well supported by the hot permeability of the systems tested. Adding jute fibers increased permeability of UHPC at high temperatures due to the shrinkage of fibers, and as a result, creation of interfacial gaps between the fiber and cement matrix. This also highlighted the importance of percolation of fibers in the matrix.

- (2) The compressive strength of UHPC increased after exposure to 200 °C and then decreased when temperature was greater than 400 °C. With 3 kg/m³ of jute fibers, UHPC had a similar compressive strength behavior as UHPC-C at all temperatures. Adding 5 kg/m³ and 10 kg/m³ of jute fibers, a reduction in compressive strength was seen at room temperature. At higher temperatures (>400 °C), the higher the fiber dosage, the shaper decline the compressive strength experienced.
- (3) The addition of jute fibers reduced flexural strength of UHPC at room temperature but increased flexural strength of UHPC at high temperature. UHPC with jute fiber showed a lower decreasing rate on flexural strength with increasing temperature.
- (4) Exposing the UHPC/jute fibers systems to various accelerated weathering conditions has little effect on their compressive and flexural strength values. Spalling resistance of UHPC with 10 kg/m³ jute fiber samples was not influenced by accelerated weathering conditions.

CHAPTER 7 CONCLUSIONS AND FUTURE WORK

7.1 Conclusions

In this research work, spalling resistance of UHPC with fibers is investigated. For this purpose, experimental studies are conducted by adopting a range of analytical, physical and microscopic characterization techniques. The mechanical properties, permeability, and microstructure of UHPC are investigated thoroughly. This work aims to understand the mechanism of polymer fibers and facilitate the optimization for spalling prevention purpose, without too many trial-and-error experiments. In addition, the potential of using natural fibers to prevent spalling is also investigated. Main conclusions of this research work are summarized as follows:

Mechanism of preventing spalling of UHPC by polymer fibers.

Inclusion of PP fibers increases the gas permeability of UHPC at high temperature, and hence, improves the spalling resistance of UHPC. The increase in permeability mainly occurs from around 150 °C, which is below the melting point of PP fibers (around 170 °C). Further increase in permeability at 200 °C or even 300 °C is relatively small. The empty channels left by PP fibers do not contribute significantly to the permeability of UHPC. The melting of fibers and the creation of empty channels are not the reasons for the increase in permeability of UHPC.

The increase in permeability of UHPC before fiber melting is attributed to the formation of interconnecting network of microcracks. The high thermal mismatch between embedded fibers and cement matrix is critical to forming microcracks in UHPC. Thermal expansion of PP fibers creates microcracks in UHPC before melting of fibers. The interconnecting networks of microcracks enhance the permeability and

improve spalling resistance of UHPC.

LLDPE and PA66 fibers can improve spalling resistance of UHPC due to their high thermal expansion before fiber melting, while UHMWPE and PET fibers do not improve spalling resistance of UHPC since their thermal expansion is relatively small. Melting point of fiber is not critical to spalling prevention of UHPC.

Spatial distribution of PP fibers in UHPC and its effect on spalling prevention

The formation of interconnecting network of microcracks is related to fiber distribution, i.e. connectivity, distance, and percolation. At 150 °C, a significant increase in permeability occurs in samples with percolation of fibers. The percolation of fibers is beneficial in forming a percolating interconnecting network of microcracks at this temperature (before fiber melting). This results in an increase in permeability, thus enhancing spalling resistance of UHPC. The number of percolating microcracks is related to the number of percolating fibers. Therefore, the number of fibers that are connected to percolating fiber cluster is crucial to achieving high permeability and enhancing spalling resistance.

Interconnecting network of microcracks is easier to be created under high connectivity and small distance of fiber. Increasing fiber length, reducing fiber diameter (in simple terms, higher aspect ratio) and increasing fiber dosage result in an increase in fiber connectivity and a decrease in inter-fiber distance, which leads to an increase in permeability.

Adding fibers affects flowability and compressive strength of UHPC. Longer fiber and higher fiber dosage can reduce flowability. Lowering the flowability increases air bubble content in UHPC. However, the changes in air bubble content caused by adding fibers do not affect spalling resistance of UHPC. A very high fiber dosage (12

kg/m³ in this study) can cause a significant decrease in compressive strength.

Critical fiber dosage for spalling prevention of UHPC

A percolation model is developed to estimate percolation and connectivity of fiber/crack system in UHPC. Percolation threshold of the system reduces with increasing aspect (l_f/d_f) ratio of fibers. When PP fiber dosages are lower than the percolation threshold of the system, the addition of PP fibers cannot increase permeability or enhance spalling resistance of UHPC significantly.

An approach based on the Kozney-Carmon equation is proposed to predict the permeability of UHPC with PP fibers of a dosage higher than percolation threshold. A semi-empirical equation is proposed to estimate the critical fiber dosages of different fiber geometries (PBN fiber dosage) for preventing spalling. The equation has been validated by a series of spalling tests. The PBN fiber dosage does not reduce at a l_f/d_f ratio > 600 , but it increases significantly at a low l_f/d_f ratio < 150 . PP fibers with a l_f/d_f ratio between 300 to 600 are recommended for spalling prevention of UHPC. Furthermore, at a constant fiber dosage, the effect of fiber length on permeability becomes limited at a fiber length larger than 18 mm.

Natural fibers on spalling prevention of UHPC

Adding jute fibers can increase permeability and improve spalling resistance of UHPC. Shrinkage of jute fibers at high temperature creates space at the interface between the natural fiber and the cement paste, hence increasing permeability. Percolation of jute fiber system is required for increasing permeability. A dosage of 10 kg/m³ of jute fibers is sufficient to prevent UHPC from spalling in this study.

However, adding a high dosage of jute fibers (above 5 kg/m³) reduces compressive

strength of UHPC at ambient and high temperature. At 400 °C and above, the greater the fiber dosage, the more significant the decrease in compressive strength is. Jute fibers reduce flexural strength of UHPC at room temperature but increase flexural strength at high temperature. Within the range from 200 °C to 600 °C, UHPC with jute fibers shows a lower decreasing rate on flexural strength with increasing temperature.

Accelerated weathering curing has a limited effect on compressive strength and flexural strength of UHPC with jute fibers. Spalling resistance of UHPC with 10 kg/m³ of jute fibers can be maintained after accelerated weathering curing.

7.2 Future work

Although the research work presented here contributes to new knowledge on spalling prevention of UHPC material, there are still many aspects to be studied, as follows:

- (1) A 3D characterization on microcracks in UHPC will be very useful in improving the understanding on the formation of interconnecting network. This can supplement and enhance the findings in CHAPTERS 3 and 4. However, the required high resolution of tomography versus the size of sample is a major limitation currently.
- (2) Spalling test results could be affected by many factors, such as testing method, heating rate, specimen dimensions, aggregate size and type, dispersion of fibers, and steel fibers. The method proposed in CHAPTER 5 should be further developed by considering some of these parameters.
- (3) Degradation of natural fibers in alkaline condition is a critical issue to be investigated further. It is known that mercerization of natural fibers for shorter

periods of 8-12 h will increase the amount of cellulose II (anti-parallel chain conformation) at the expense of cellulose I (parallel chain arrangement), which is an irreversible polymorphic transformation. This will also be associated with a huge drop in the percentage of crystallinity of fibers, as well as size reduction and roughening of the surface of the fibers. It is important to understand how these different parameters of natural fibers affect the properties of UHPC.

- (4) Duration of accelerated weathering curing in this study requires further studies for assessing the long-term durability of natural fibers. More thorough and in-depth investigations on accelerated weathering of natural fibers cast in UHPC should be carried out. Meanwhile, a reliable method to assess the durability of natural fiber embedded concrete should be developed as well.
- (5) The spalling tests conducted in this thesis are all based on small cylinder samples, which is at material level. It is important to understand the spalling behavior of UHPC with fibers at structural level. Therefore, it is necessary to conduct fire tests on UHPC members, such as columns, beams, and slabs. Different parameters, including fibers, size of specimen and external loading, should be considered in the fire tests.

REFERENCES

- Abid, M., X. Hou, W. Zheng and R. R. Hussain (2017). "High temperature and residual properties of reactive powder concrete – A review." Construction and Building Materials **147**: 339-351.
- Abràmoff, M. D., P. J. Magalhães and S. J. Ram (2004). "Image processing with ImageJ." Biophotonics international **11**(7): 36-42.
- Akhavan, A., S.-M.-H. Shafaatian and F. Rajabipour (2012). "Quantifying the effects of crack width, tortuosity, and roughness on water permeability of cracked mortars." Cement and Concrete Research **42**(2): 313-320.
- Alarcon-Ruiz, L., G. Platret, E. Massieu and A. Ehrlacher (2005). "The use of thermal analysis in assessing the effect of temperature on a cement paste." Cement and Concrete Research **35**(3): 609-613.
- Alonso, M. C., V. Flor-Laguna and M. Sanchez (2013). "Microstructural response of polypropylene fibres at high temperature to protect concrete from spalling." MATEC Web of Conferences **6**: 02001.
- ASTM (2014). C469 / C469M-14 Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression. West Conshohocken, PA,, ASTM International.
- ASTM (2015). C1437-15, Standard test method for flow of hydraulic cement mortar. West Conshohocken, PA, ASTM International.
- ASTM (2016). C109 / C109M-16a, Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens) West Conshohocken, PA, ASTM International.
- ASTM (2016). G154-16, Standard Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Nonmetallic Materials. West Conshohocken, PA, ASTM International.
- ASTM (2017). C496 / C496M-17, Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens. West Conshohocken, PA,, ASTM International.
- ASTM (2017). E399-17, Standard Test Method for Linear-Elastic Plane-Strain Fracture Toughness K_{Ic} of Metallic Materials. West Conshohocken, PA, ASTM International.
- Bangi, M. R. and T. Horiguchi (2011). "Pore pressure development in hybrid fibre-reinforced high strength concrete at elevated temperatures." Cement and Concrete Research **41**(11): 1150-1156.

- Bangi, M. R. and T. Horiguchi (2012). "Effect of fibre type and geometry on maximum pore pressures in fibre-reinforced high strength concrete at elevated temperatures." Cement and Concrete Research **42**(2): 459-466.
- Bažant, Z. P. and B. H. Oh (1983). "Crack band theory for fracture of concrete." Matériaux et Construction **16**(3): 155-177.
- BEA-TT & RAIB (2010). Technical investigation report concerning the fire on Eurotunnel freight shuttle 7412 on 11 September 2008, La Défense cedex, Paris, France / Derby, United Kingdom: Bureau d'enquêtes sur les accidents de transport terrestre / Rail Accident Investigation Branch.
- Behnood, A. and H. Ziari (2008). "Effects of silica fume addition and water to cement ratio on the properties of high-strength concrete after exposure to high temperatures." Cement and Concrete Composites **30**(2): 106-112.
- Bentz, D. P. (2000). "Fibers, percolation, and spalling of high-performance concrete." ACI Materials Journal **97**(3): 351-359.
- Bordelon, A. C. and J. R. Roesler (2014). "Spatial distribution of synthetic fibers in concrete with X-ray computed tomography." Cement and Concrete Composites **53**: 35-43.
- Bošnjak, J., J. Ožbolt and R. Hahn (2013). "Permeability measurement on high strength concrete without and with polypropylene fibers at elevated temperatures using a new test setup." Cement and Concrete Research **53**: 104-111.
- Bossa, N., P. Chaurand, J. Vicente, D. Borschneck, C. Levard, O. Aguerre-Chariol and J. Rose (2015). "Micro- and nano-X-ray computed-tomography: A step forward in the characterization of the pore network of a leached cement paste." Cement and Concrete Research **67**: 138-147.
- Boström, L. and R. Jansson (2008). Self-compacting concrete exposed to fire.
- British Standards Institution (2005). BS EN 1992-1-2:2004 Eurocode 2: Design of concrete structures — Part 1-2: General rules — Structural fire design. London, United Kingdom, BSI Group.
- British Standards Institution (2012). BS-EN 494:2012, Fibre-cement profiled sheets and fittings - Product specification and test methods, BSI.
- Bruker-microCT (2013). Manual for Bruker-microCT CT-Analyser v. 1.13.
- Carman, P. C. (1939). "Permeability of saturated sands, soils and clays." The Journal of Agricultural Science **29**(2): 262-273.

Channel Tunnel Safety Authority (1997). Inquiry into the fire on heavy goods vehicle shuttle 7539 on 18 November 1996, Stationery Office.

Chen, B. and J. Liu (2004). "Residual strength of hybrid-fiber-reinforced high-strength concrete after exposure to high temperatures." Cement and Concrete Research **34**(6): 1065-1069.

Chen, X.-T., T. Rougelot, C. A. Davy, W. Chen, F. Agostini, F. Skoczylas and X. Bourbon (2009). "Experimental evidence of a moisture clog effect in cement-based materials under temperature." Cement and Concrete Research **39**(12): 1139-1148.

Consolazio, G. R., M. C. McVay and J. W. Rish III (1998). "Measurement and prediction of pore pressures in saturated cement mortar subjected to radiant heating." Materials Journal **95**(5): 525-536.

de Larrard, F. and T. Sedran (1994). "Optimization of ultra-high-performance concrete by the use of a packing model." Cement and Concrete Research **24**(6): 997-1009.

Delale, F. (1988). "Critical fiber size for microcrack suppression in ceramic-fiber/ceramic-matrix composites." Engineering Fracture Mechanics **31**(1): 145-155.

Delale, F. and H. Boduroglu (1991). "Effect of thermal expansion anisotropy on microcracking in ceramic composites." Engineering Fracture Mechanics **39**(1): 45-60.

der Heijden, G. H. A. v., L. Pel and O. C. G. Adan (2012). "Fire spalling of concrete, as studied by NMR." Cement and Concrete Research **42**(2): 265-271.

Dias, W., G. Khoury and P. Sullivan (1990). "Shrinkage of hardened cement paste at temperatures up to 670 C (1238 F)." ACI Materials Journal **87**(Title No. 87-M22).

Diederichs, U., U. M. Jumppanen and U. Schneide (1995). High Temperature Properties and Spalling Behaviour of High Strength Concrete. Fourth Weimar Workshop on High Performance Concrete: Material Properties and Design, Weimar, Germany, Hochschule für Architektur und Bauwesen.

Diederichs, V., J. Spitzner, M. Sandvik, B. Kepp and M. Gillen (1993). The behaviour of high-strength lightweight aggregate concrete at elevated temperature. Symposium on utilization of high strength concrete. Norway, Norwegian Concrete Association Lillehammer: 1046-1053.

Ding, Y., C. Zhang, M. Cao, Y. Zhang and C. Azevedo (2016). "Influence of different fibers on the change of pore pressure of self-consolidating concrete exposed to fire." Construction and Building Materials **113**: 456-469.

Dougill, J. W. (1971). The effects of high temperature on the strength of concrete with reference to

thermal spalling, Imperial College London (University of London).

Dwaikat, M. B. and V. K. R. Kodur (2010). "Fire Induced Spalling in High Strength Concrete Beams." Fire Technology **46**(1): 251-274.

Fares, H., S. Remond, A. Noumowe and A. Cousture (2010). "High temperature behaviour of self-consolidating concrete Microstructure and physicochemical properties." Cement and Concrete Research **40**(3): 488-496.

Felicetti, R., F. Lo Monte and P. Pimienta (2017). "A new test method to study the influence of pore pressure on fracture behaviour of concrete during heating." Cement and Concrete Research **94**: 13-23.

Folias, E. S. and M. Hohn (1998). "Predicting crack initiation in composite material systems due to a thermal expansion mismatch." International Journal of Fracture **93**(1): 335-349.

Fu, Y. and L. Li (2011). "Study on mechanism of thermal spalling in concrete exposed to elevated temperatures." Materials and structures **44**(1): 361-376.

Gary, M. (1918). Brandproben an Eisenbetonbauten: ausgeführt im königl. Materialprüfungsamt zu Berlin-Lichterfelde-West in d. Jahren 1914 u. 1915, Ernst.

Grassl, P., H. S. Wong and N. R. Buenfeld (2010). "Influence of aggregate size and volume fraction on shrinkage induced micro-cracking of concrete and mortar." Cement and Concrete Research **40**(1): 85-93.

Grattan-Bellew, P. (1996). "Microstructural investigation of deteriorated Portland cement concretes." Construction and Building Materials **10**(1): 3-16.

Gu, H., J. Wang and C. Yu (2016). "Three-dimensional modeling of percolation behavior of electrical conductivity in segregated network polymer nanocomposites using Monte Carlo method." Advances in Materials **5**(1): 1-8.

Guerrieri, M., J. Sanjayan and F. Collins (2008). "Effect of Slag on the Performance of Concretes in Hydrocarbon Fire." ACI SP225-02: Designing Concrete Structure for Fire Safety, ACI-AMS Committee 216: Fire Resistance and Fire Protection of Structures **255**: 23-46.

Hager, I. and T. Tracz (2010). "The impact of the amount and length of fibrillated polypropylene fibres on the properties of HPC exposed to high temperature." Archives of Civil Engineering **56**(1): 57-68.

Hager, I., T. Zdeb and K. Krzemien (2013). The impact of the amount of polypropylene fibres on spalling behaviour and residual mechanical properties of Reactive Powder Concretes. Concrete

Spalling due to Fire Exposure: 3rd International Workshop, Paris, France, EDP Sciences.

Han, C.-G., M.-C. Han and Y.-S. Heo (2009). "Improvement of residual compressive strength and spalling resistance of high-strength RC columns subjected to fire." Construction and Building Materials **23**(1): 107-116.

Handoo, S., S. Agarwal and S. Agarwal (2002). "Physicochemical, mineralogical, and morphological characteristics of concrete exposed to elevated temperatures." Cement and Concrete Research **32**(7): 1009-1018.

Harmathy, T. (1965). Effect of moisture on the fire endurance of building elements. Moisture in materials in relation to fire tests, ASTM International.

Harmathy, T. Z. (1966). "Experimental study on moisture and fire endurance." Fire Technology **2**(1): 52-59.

He, S., J. Qiu, J. Li and E.-H. Yang (2017). "Strain hardening ultra-high performance concrete (SHUHPC) incorporating CNF-coated polyethylene fibers." Cement and Concrete Research **98**: 50-60.

Heinz, D., F. Dehn and L. Urbonas (2004). Fire resistance of ultra-high performance concrete (UHPC)—testing of laboratory samples and columns under load. International Symposium on High Performance Concrete; Ultra high performance concrete. E. F. M. Schmidt, C. Geisenhanslüke. Kassel, Germany, Kassel University Press: 703-715.

Henderson, N., J. C. Brêttas and W. F. Sacco (2010). "A three-parameter Kozeny–Carman generalized equation for fractal porous media." Chemical Engineering Science **65**(15): 4432-4442.

Heo, Y.-S., C.-G. Han and K.-M. Kim (2014). "Combined fiber technique for spalling protection of concrete column, slab and beam in fire." Materials and Structures **48**(10): 3377-3390.

Heo, Y.-S., J. G. Sanjayan, C.-G. Han and M.-C. Han (2010). "Critical parameters of nylon and other fibres for spalling protection of high strength concrete in fire." Materials and Structures **44**(3): 599-610.

Heo, Y.-S., J. G. Sanjayan, C.-G. Han and M.-C. Han (2010). "Synergistic effect of combined fibers for spalling protection of concrete in fire." Cement and Concrete Research **40**(10): 1547-1554.

Heo, Y.-S., J. G. Sanjayan, C.-G. Han and M.-C. Han (2011). "Limited effect of diameter of fibres on spalling protection of concrete in fire." Materials and Structures **45**(3): 325-335.

- Heo, Y.-S., J. G. Sanjayan, C.-G. Han and M.-C. Han (2012). "Relationship between inter-aggregate spacing and the optimum fiber length for spalling protection of concrete in fire." Cement and Concrete Research **42**(3): 549-557.
- Hertz, K. (1984). "Explosion of silica-fume concrete." Fire safety journal **8**(1): 77-77.
- Hertz, K. D. (1992). "Danish investigations on silica fume concretes at elevated temperatures." Materials Journal **89**(4): 345-347.
- Hertz, K. D. (2003). "Limits of spalling of fire-exposed concrete." Fire Safety Journal **38**(2): 103-116.
- Hertz, K. D. (2005). "Concrete strength for fire safety design." Magazine of Concrete Research **57**(8): 445-453.
- Hillerborg, A., M. Modr and P. E. Petersson (1976). "Analysis of crack formation and crack growth in concrete by means of fracture mechanics and finite elements." Cement and Concrete Research **6**(6): 773-781.
- Ho, S. and Z. Suo (1992). "Microcracks tunneling in brittle matrix composites driven by thermal expansion mismatch." Acta Metallurgica et Materialia **40**(7): 1685-1690.
- Hsieh, J. (2009). Computed tomography : principles, design, artifacts, and recent advances, Bellingham, Wash. : SPIE Press ; Hoboken, N.J. : Wiley Interscience, c2009.2nd ed.
- Jacobs, F. and F. Hunkeler (2006). Non destructive testing of the concrete cover–evaluation of permeability test data. International RILEM Workshop on Performance Based Evaluation and Indicators for Concrete Durability, Madrid, Spain, RILEM Publications.
- Jansson, R. (2013). Fire Spalling of Concrete: Theoretical and Experimental Studies. Ph.D. Ph.D. Thesis, KTH Royal Institute of Technology in Stockholm.
- Jansson, R. and L. Bostrm (2008). Spalling of concrete exposed to fire. Sweden, SP Technical Research Institute of Sweden.
- Jumppanen, U.-M. (1989). "Effect of strength on fire behaviour of concrete." Nordic concrete research(8): 116-127.
- Kahanji, C., F. Ali and A. Nadjai (2016). "Explosive spalling of ultra-high performance fibre reinforced concrete beams under fire." Journal of Structural Fire Engineering **7**(4): 328-348.
- Kalifa, P., G. Chene and C. Galle (2001). "High-temperature behaviour of HPC with polypropylene

- fibres: From spalling to microstructure." Cement and concrete research **31**(10): 1487-1499.
- Kalifa, P., F.-D. Menneteau and D. Quenard (2000). "Spalling and pore pressure in HPC at high temperatures." Cement and Concrete Composites **30**: 1915-1927.
- Khoury, G. A. (2000). "Effect of fire on concrete and concrete structures." Progress in Structural Engineering and Materials **2**(4): 429-447.
- Khoury, G. A. (2008). "Polypropylene fibres in heated concrete. Part 2: Pressure relief mechanisms and modelling criteria." Magazine of Concrete Research **60**(3): 189-204.
- Khoury, G. A. and Y. Anderberg (2000). "Concrete spalling review." Fire Safety Design **60**: 5-12.
- Khoury, G. A. and B. Willoughby (2008). "Polypropylene fibres in heated concrete. Part 1: Molecular structure and materials behaviour." Magazine of Concrete Research **60**(2): 125-136.
- Kim, K. Y., T. S. Yun and K. P. Park (2013). "Evaluation of pore structures and cracking in cement paste exposed to elevated temperatures by X-ray computed tomography." Cement and Concrete Research **50**: 34-40.
- Klingsch, E., A. Frangi and M. Fontana (2013). Explosive spalling of concrete in fire, Institut für Baustatik und Konstruktion, ETH Zürich.
- Klingsch, E. W. H. (2014). Explosive spalling of concrete in fire Ph.D. Thesis, Swiss Federal Institute of Technology in Zurich (ETHZ).
- Knack, I., E. Koenders and F. Dehn (2011). The use of PP fibers in tunnel construction to avoid explosive concrete spalling in case of fire. New test results for the clarification of the mode of action. Proceedings of the 2nd International RILEM Workshop on Concrete Spalling due to Fire Exposure, Delft, The Netherlands.
- Kodur, V. (2000). Spalling in high strength concrete exposed to fire—concerns, causes, critical parameters and cures. Proceedings, ASCE Structures Congress, Philadelphia, PA.
- Kodur, V. (2014). "Properties of Concrete at Elevated Temperatures." ISRN Civil Engineering **2014**: 1-15.
- Kodur, V. and R. McGrath (2003). "Fire endurance of high strength concrete columns." Fire technology **39**(1): 73-87.
- Kodur, V. K. R. and L. Phan (2007). "Critical factors governing the fire performance of high strength

- concrete systems." Fire Safety Journal **42**(6-7): 482-488.
- Kozeny, J. (1927). "Uber kapillare leitung der wasser in boden." Royal Academy of Science, Vienna, Proc. Class I **136**: 271-306.
- Lai, J., S. Xu, C. Yang, X. Guo and Y. zhu (2013). "Influence of polyvinyl alcohol fibers on the properties of ultra high performance concrete at high temperature (in Chinese)." Journal of Nanjing University of Science and Technology **37**(4): 633-639.
- Latief, F. D. E. and U. Fauzi (2012). "Kozeny–Carman and empirical formula for the permeability of computer rock models." International Journal of Rock Mechanics and Mining Sciences **50**: 117-123.
- Lee, G., D. Han, M.-C. Han, C.-G. Han and H.-J. Son (2012). "Combining polypropylene and nylon fibers to optimize fiber addition for spalling protection of high-strength concrete." Construction and Building Materials **34**: 313-320.
- Lee, J. H., Y. S. Sohn and S. H. Lee (2012). "Fire resistance of hybrid fibre-reinforced, ultra-high-strength concrete columns with compressive strength from 120 to 200 MPa." Magazine of Concrete Research **64**(6): 539-550.
- Lee, K. K. (2008). Evaluation of concrete behavior under high temperature. 3315814 Ph.D., University of Colorado at Boulder.
- Legland, D. (2018). "geom3d (<https://www.mathworks.com/matlabcentral/fileexchange/24484-geom3d>), MATLAB Central File Exchange. Retrieved December 22, 2018."
- Leguillon, D. (2002). "Strength or toughness? A criterion for crack onset at a notch." European Journal of Mechanics-A/Solids **21**(1): 61-72.
- Li, J. and S.-L. Zhang (2009). "Finite-size scaling in stick percolation." Physical Review E **80**(4): 040104.
- Li, Y. (2018). Material properties and explosive spalling of ultra-high performance concrete in fire. PhD, Nanyang Technological University.
- Li, Y., P. Pimienta, N. Pinoteau and K. H. Tan (2019). "Effect of aggregate size and inclusion of polypropylene and steel fibers on explosive spalling and pore pressure in ultra-high-performance concrete (UHPC) at elevated temperature." Cement and Concrete Composites **99**: 62-71.
- Li, Y. and K. H. Tan (2016). Effects of Polypropylene and Steel Fibers on Permeability of Ultra-high Performance Concrete at Hot State. Structures in Fire (Proceedings of the Ninth International

Conference). M. E. M. Garlock and V. K. R. Kodur. Princeton University, DEStech Publications: 145-152.

Li, Y., K. H. Tan and E.-H. Yang (2018). "Influence of aggregate size and inclusion of polypropylene and steel fibers on the hot permeability of ultra-high performance concrete (UHPC) at elevated temperature." Construction and Building Materials **169**: 629-637.

Li, Y., K. H. Tan and E.-H. Yang (2019). "Synergistic effects of hybrid polypropylene and steel fibers on explosive spalling prevention of ultra-high performance concrete at elevated temperature." Cement and Concrete Composites **96**: 174-181.

Li, Y., Y. Zhang, E.-H. Yang and K. H. Tan (2019). "Effects of geometry and fraction of polypropylene fibers on permeability of ultra-high performance concrete after heat exposure." Cement and Concrete Research **116**: 168-178.

Liang, X., C. Wu, Y. Su, Z. Chen and Z. Li (2018). "Development of ultra-high performance concrete with high fire resistance." Construction and Building Materials **179**: 400-412.

Liu, J.-C., K. H. Tan and Y. Yao (2018). "A new perspective on nature of fire-induced spalling in concrete." Construction and Building Materials **184**: 581-590.

Liu, J.-C., K. H. Tan and D. Zhang (2017). "Multi-response optimization of post-fire performance of strain hardening cementitious composite." Cement and Concrete Composites **80**: 80-90.

Liu, X., G. Ye, G. De Schutter, Y. Yuan and L. Taerwe (2008). "On the mechanism of polypropylene fibres in preventing fire spalling in self-compacting and high-performance cement paste." Cement and Concrete Research **38**(4): 487-499.

Lottman, B. B. G. (2017). The spalling mechanism of fire exposed concrete.

Lu, T. C., A. G. Evans, R. J. Hecht and R. Mehrabian (1991). "Toughening of MoSi₂ with a ductile (niobium) reinforcement." Acta Metallurgica et Materialia **39**(8): 1853-1862.

Lu, T. C., J. Yang, Z. Suo, A. G. Evans, R. Hecht and R. Mehrabian (1991). "Matrix cracking in intermetallic composites caused by thermal expansion mismatch." Acta Metallurgica et Materialia **39**(8): 1883-1890.

Ma, H., S. Qian, Z. Zhang, Z. Lin and V. C. Li (2015). "Tailoring Engineered Cementitious Composites with local ingredients." Construction and Building Materials **101**: 584-595.

Ma, J. (2015). "Review of permeability evolution model for fractured porous media." Journal of Rock

Mechanics and Geotechnical Engineering 7(3): 351-357.

Ma, Q., R. Guo, Z. Zhao, Z. Lin and K. He (2015). "Mechanical properties of concrete at high temperature—A review." Construction and Building Materials 93: 371-383.

Malhotra, H. (1984). Spalling of concrete in fires. London, UK, CIRIA.

Maluk, C., L. Bisby and G. P. Terrasi (2017). "Effects of polypropylene fibre type and dose on the propensity for heat-induced concrete spalling." Engineering Structures 141: 584-595.

Mathworks (2018). "Global Optimization Toolbox: User's Guide (R2018a). March 2018. From https://www.mathworks.com/help/pdf_doc/gads/gads_tb.pdf."

Mavko, G. and A. Nur (1997). "The effect of a percolation threshold in the Kozeny-Carman relation." Geophysics 62(5): 1480-1482.

Mazzucco, G., C. E. Majorana and V. A. Salomoni (2015). "Numerical simulation of polypropylene fibres in concrete materials under fire conditions." Computers & Structures 154: 17-28.

Mehta, P. K. and P. J. M. Monteiro (2006). Concrete: Structure, properties and materials. New York, United States, McGraw-Hill Education

Meyer-Ottens, C. (1972). Zur Frage der Abplatzungen an Betonbauteilen aus Normalbeton bei Brandbeanspruchung [Spalling of normal-strength concrete structures under fire loading]. Ph.D, Braunschweig University of Technology.

Mindeguia, J.-C., P. Pimienta, A. Noumowé and M. Kanema (2010). "Temperature, pore pressure and mass variation of concrete subjected to high temperature — Experimental and numerical discussion on spalling risk." Cement and Concrete Research 40(3): 477-487.

Missemer, L., E. Ouedraogo, Y. Malecot, C. Clergue and D. Rogat (2019). "Fire spalling of ultra-high performance concrete: From a global analysis to microstructure investigations." Cement and Concrete Research 115: 207-219.

Missemer, L., E. Ouedraogo, Y. Malecot, D. Rogat and C. Clergue (2010). Effect of polymer fibres inclusion in fire spalling of ultra-high performance concrete. 7th International Conference on Fracture Mechanics Concrete and Concrete Structures, Jeju, Korea.

Missemer, L., E. Ouedraogo, Y. Malecot, D. Rogat and C. Clergue (2010). Spalling under fire of ultra-high performance fibre concrete: Effect of polymer fibers. Sixth International Conference on Concrete under Severe Conditions: Environment and Loading.

- Netinger Grubeša, I., B. Marković, A. Gojević and J. Brdarić (2018). "Effect of hemp fibers on fire resistance of concrete." Construction and Building Materials **184**: 473-484.
- Neville, A. M. (2011). Properties of concrete. Harlow, United Kingdom, Pearson Education Limited.
- Newman, M. E. and R. M. Ziff (2001). "Fast Monte Carlo algorithm for site or bond percolation." Physical Review E **64**(1): 016706.
- Nguyen, T. N. H., K.-H. Tan and T. Kanda (2019). "Investigations on web-shear behavior of deep precast, prestressed concrete hollow core slabs." Engineering Structures **183**: 579-593.
- Noumowe, A. (2005). "Mechanical properties and microstructure of high strength concrete containing polypropylene fibres exposed to temperatures up to 200 °C." Cement and Concrete Research **35**(11): 2192-2198.
- Noumowe, A. N., R. Siddique and G. Debicki (2009). "Permeability of high-performance concrete subjected to elevated temperature (600°C)." Construction and Building Materials **23**(5): 1855-1861.
- Ozawa, M., Z. Bo, J. Kawaguchi and Y. Uchida (2013). Preventive effect on spalling of UFC using jute fiber at high temperature. 3rd International Workshop on Concrete Spalling due to Fire Exposure. Paris, France. **6**: 02006.
- Ozawa, M., Z. Bo, Y. Uchida and H. Morimoto (2014). "Preventive Effects of Fibers on Spalling of UFC at High Temperatures." Journal of Structural Fire Engineering **5**(3): 229-238.
- Ozawa, M. and H. Morimoto (2014). "Effects of various fibres on high-temperature spalling in high-performance concrete." Construction and Building Materials **71**: 83-92.
- Ozawa, M., S. Subedi Parajuli, Y. Uchida and B. Zhou (2019). "Preventive effects of polypropylene and jute fibers on spalling of UHPC at high temperatures in combination with waste porous ceramic fine aggregate as an internal curing material." Construction and Building Materials **206**: 219-225.
- Ozawa, M., S. Uchida, T. Kamada and H. Morimoto (2012). "Study of mechanisms of explosive spalling in high-strength concrete at high temperatures using acoustic emission." Construction and Building Materials **37**: 621-628.
- Peng, G.-F. and Z.-S. Huang (2008). "Change in microstructure of hardened cement paste subjected to elevated temperatures." Construction and Building Materials **22**(4): 593-599.
- Peng, G. and J. Yang (2017). "Residual Mechanical Properties and Explosive Spalling Behavior of Ultra- High- Strength Concrete Exposed to High Temperature." Journal of Harbin Institute of

Technology (New Series) **24**(4): 62-70.

Persson, B. (2004). "Fire resistance of self-compacting concrete, SCC." Materials and structures **37**(9): 575-584.

Phan, L. T. (1996). Fire performance of high-strength concrete: A report of the state-of-the art.

Phan, L. T. (2002). High-strength concrete at high temperature-An Overview. Utilization of High Strength/High Performance Concrete, 6th International Symposium. Proceedings.

Pierre Pimienta, J.-C. M., Alain Simon, Mouloud Behloul, Roberto Felicetti, Patrick Bamonte, Pietro G. Gambarova (2012). Literature Review on the Behaviour of UHPFRC at High Temperature. 3rd International Symposium on UHPC and Nanotechnology for High Performance Construction Materials. E. F. M. Schmidt, C. Glotzbach, S. Fröhlich, S. Piotrowski. Kassel, German, Kassel university press.

Pimienta, P., J.-C. Mindeguia, A. Simon and M. Behloul (2009). "Behaviour of UHPFRC at high temperatures." Designing and Building with UHPFRC: State of the Art and Development.

Pistol, K. (2016). "Wirkungsweise von Polypropylen-Fasern in brandbeanspruchtem Hochleistungsbeton."

Pistol, K., F. Weise, B. Meng, U. Schneider, E. Koenders and F. Dehn (2011). The mode of action of polypropylene fibres in high performance concrete at high temperatures. Proceedings of the 2nd International RILEM Workshop on Concrete Spalling Due to Fire Exposure, Delft, The Netherlands.

Poon, C.-S., S. Azhar, M. Anson and Y.-L. Wong (2001). "Comparison of the strength and durability performance of normal- and high-strength pozzolanic concretes at elevated temperatures." Cement and Concrete Research **31**(9): 1291-1300.

Qomi, M. A., K. J. Krakowiak, M. Bauchy, K. Stewart, R. Shahsavari, D. Jagannathan, D. B. Brommer, A. Baronnet, M. J. Buehler and S. Yip (2014). "Combinatorial molecular optimization of cement hydrates." Nature communications **5**.

Ranade, R., V. C. Li, M. D. Stults, W. F. Heard and T. S. Rushing (2013). "Composite Properties of High-Strength, High-Ductility Concrete." ACI Materials Journal **110**(4).

Ranade, R., V. C. Li, M. D. Stults, T. S. Rushing, J. Roth and W. F. Heard (2013). "Micromechanics of high-strength, high-ductility concrete." ACI Materials Journal **110**(4): 375.

Rashad, A. M., Y. Bai, P. A. M. Basheer, N. C. Collier and N. B. Milestone (2012). "Chemical and mechanical stability of sodium sulfate activated slag after exposure to elevated temperature." Cement

and Concrete Research **42**(2): 333-343.

Rashad, A. M. and S. R. Zeedan (2012). "A preliminary study of blended pastes of cement and quartz powder under the effect of elevated temperature." Construction and Building Materials **29**: 672-681.

Ray, D., B. Sarkar, R. Basak and A. Rana (2002). "Study of the thermal behavior of alkali - treated jute fibers." Journal of Applied Polymer Science **85**(12): 2594-2599.

Richard, P. and M. Cheyrezy (1995). "Composition of reactive powder concretes." Cement and Concrete Research **25**(7): 1501-1511.

Ringot, E. and A. Bascoul (2001). "About the analysis of microcracking in concrete." Cement and concrete composites **23**(2): 261-266.

Roy, A., S. Chakraborty, S. P. Kundu, R. K. Basak, S. B. Majumder and B. Adhikari (2012). "Improvement in mechanical properties of jute fibres through mild alkali treatment as demonstrated by utilisation of the Weibull distribution model." Bioresour Technol **107**: 222-228.

Saito, H. (1966). "Explosive spalling of prestressed concrete in fire." Bulletin of Japan Association for Fire Science and Engineering **15**(2): 23-30.

Sanchayan, S. and S. Foster (2015). "High temperature behaviour of hybrid steel–PVA fibre reinforced reactive powder concrete." Materials and Structures: 1-14.

Sertmehmetoglu, Y. (1977). On a mechanism of spalling of concrete under fire conditions, University of London.

Shi, C., Z. Wu, J. Xiao, D. Wang, Z. Huang and Z. Fang (2015). "A review on ultra high performance concrete: Part I. Raw materials and mixture design." Construction and Building Materials **101**: 741-751.

Shorter, G. and T. Harmathy (1961). "Discussion on the fire resistance of prestressed concrete beams." Proceedings of the Institution of Civil Engineers **20**(2): 313-315.

Sideris, K. K., P. Manita and E. Chaniotakis (2009). "Performance of thermally damaged fibre reinforced concretes." Construction and Building Materials **23**(3): 1232-1239.

Siemon, M. and J. Zehfuß (2015). Experimental analysis and numerical simulation of Ultra High Performance Concrete (UHPC) members in case of fire. Applications of Structural Fire Engineering. Dubrovnik, Croatia.

Silva, F. d. A., B. Mobasher, C. Soranakom and R. D. T. Filho (2011). "Effect of fiber shape and morphology on interfacial bond and cracking behaviors of sisal fiber cement based composites." Cement and Concrete Composites **33**(8): 814-823.

Smith, K. and T. Atkinson (2010). Factors to consider in using PP fibres in concrete to provide explosive spalling resistance in the event of a fire. Shotcrete, CRC Press: 261-268.

So, H.-s., H.-s. Jang, J. Khulgadai and S.-y. So (2015). "Mechanical properties and microstructure of reactive powder concrete using ternary pozzolanic materials at elevated temperature." KSCE Journal of Civil Engineering **19**(4): 1050-1057.

Suhaendi, S. L. and T. Horiguchi (2006). "Effect of short fibers on residual permeability and mechanical properties of hybrid fibre reinforced high strength concrete after heat exposition." Cement and Concrete Research **36**(9): 1672-1678.

Sullivan, P. J. E. (2001). Deterioration and Spalling of High Strength Concrete Under Fire. Report for UK Health & Safety Executive, City University London.

Sun, B. and Z. Lin (2016). "Investigation on spalling resistance of ultra-high-strength concrete under rapid heating and rapid cooling." Case Studies in Construction Materials **4**: 146-153.

Tai, Y.-S., H.-H. Pan and Y.-N. Kung (2011). "Mechanical properties of steel fiber reinforced reactive powder concrete following exposure to high temperature reaching 800°C." Nuclear Engineering and Design **241**(7): 2416-2424.

Tam, C. M., V. W. Y. Tam and K. M. Ng (2012). "Assessing drying shrinkage and water permeability of reactive powder concrete produced in Hong Kong." Construction and Building Materials **26**(1): 79-89.

Tiab, D. and E. C. Donaldson (2015). Petrophysics: theory and practice of measuring reservoir rock and fluid transport properties, Gulf professional publishing.

Torrent, R. J. (1992). "A two-chamber vacuum cell for measuring the coefficient of permeability to air of the concrete cover on site." Materials and Structures **25**(6): 358-365.

Tran, V., F. Meftah, L. Izoret and M. Behloul (2013). On explicit modeling of polypropylene fiber effects on hydro-thermal behavior of heated concrete. MATEC Web of Conferences, EDP Sciences.

Wang, C., C. Yang, F. Liu, C. Wan and X. Pu (2012). "Preparation of Ultra-High Performance Concrete with common technology and materials." Cement and Concrete Composites **34**(4): 538-544.

- Wang, D., C. Shi, Z. Wu, J. Xiao, Z. Huang and Z. Fang (2015). "A review on ultra high performance concrete: Part II. Hydration, microstructure and properties." Construction and Building Materials **96**: 368-377.
- Wang, H.-L., J.-G. Dai, X.-Y. Sun and X.-L. Zhang (2016). "Characteristics of concrete cracks and their influence on chloride penetration." Construction and Building Materials **107**: 216-225.
- Wang, R., X. Gao, J. Zhang and G. Han (2018). "Spatial distribution of steel fibers and air bubbles in UHPC cylinder determined by X-ray CT method." Construction and Building Materials **160**: 39-47.
- Wei, J. and C. Meyer (2015). "Degradation mechanisms of natural fiber in the matrix of cement composites." Cement and Concrete Research **73**: 1-16.
- Weng, Y., M. Li, M. J. Tan and S. Qian (2018). "Design 3D printing cementitious materials via Fuller Thompson theory and Marson-Percy model." Construction and Building Materials **163**: 600-610.
- Wong, H. S., N. R. Buenfeld and M. K. Head (2006). "Estimating transport properties of mortars using image analysis on backscattered electron images." Cement and Concrete Research **36**(8): 1556-1566.
- Wu, Z., C. Shi and K. H. Khayat (2018). "Multi-scale investigation of microstructure, fiber pullout behavior, and mechanical properties of ultra-high performance concrete with nano-CaCO₃ particles." Cement and Concrete Composites **86**: 255-265.
- Wu, Z., H. S. Wong and N. R. Buenfeld (2015). "Influence of drying-induced microcracking and related size effects on mass transport properties of concrete." Cement and Concrete Research **68**: 35-48.
- Wypych, G. (2012). PP polypropylene. Handbook of Polymers. Oxford, Elsevier: 479-486.
- Wypych, G. (2016). Handbook of polymers, Elsevier.
- Xiong, M.-X. and J. Y. R. Liew (2015). "Spalling behavior and residual resistance of fibre reinforced Ultra-High performance concrete after exposure to high temperatures." Materiales de Construcción **65**(320): e071.
- Yan, L., N. Chouw and X. Yuan (2012). "Improving the mechanical properties of natural fibre fabric reinforced epoxy composites by alkali treatment." Journal of Reinforced Plastics and Composites **31**(6): 425-437.
- Ye, G. (2003). Experimental study and numerical simulation of the development of the microstructure and permeability of cementitious materials Ph.D. Thesis, Delft University of Technology.

- Yio, M. H. N., M. J. Mac, H. S. Wong and N. R. Buenfeld (2015). "3D imaging of cement-based materials at submicron resolution by combining laser scanning confocal microscopy with serial sectioning." Journal of Microscopy **258**(2): 151-169.
- Zeiml, M., D. Leithner, R. Lackner and H. A. Mang (2006). "How do polypropylene fibers improve the spalling behavior of in-situ concrete?" Cement and Concrete Research **36**(5): 929-942.
- Zhang, D., A. Dasari and K. H. Tan (2018). "On the mechanism of prevention of explosive spalling in ultra-high performance concrete with polymer fibers." Cement and Concrete Research **113**: 169-177.
- Zhang, H. and C. Davie (2013). "A numerical investigation of the influence of pore pressures and thermally induced stresses for spalling of concrete exposed to elevated temperatures." Fire Safety Journal **59**: 102-110.
- Zhao, J., J.-j. Zheng, G.-f. Peng and K. van Breugel (2014). "A meso-level investigation into the explosive spalling mechanism of high-performance concrete under fire exposure." Cement and concrete research **65**: 64-75.
- Zheng, W., H. Li and Y. Wang (2012). "Compressive behaviour of hybrid fiber-reinforced reactive powder concrete after high temperature." Materials & Design **41**: 403-409.
- Zheng, W., B. Luo and Y. Wang (2013). "Compressive and tensile properties of reactive powder concrete with steel fibres at elevated temperatures." Construction and Building Materials **41**: 844-851.
- Zheng, W., B. Luo and Y. Wang (2014). "Microstructure and mechanical properties of RPC containing PP fibres at elevated temperatures." Magazine of Concrete Research **66**(8): 397-408.
- Zhou, C., K. Li and X. Pang (2012). "Geometry of crack network and its impact on transport properties of concrete." Cement and Concrete Research **42**(9): 1261-1272.
- Zhou, Q. and F. Glasser (2001). "Thermal stability and decomposition mechanisms of ettringite at < 120 C." Cement and Concrete Research **31**(9): 1333-1339.
- Zhukov, V. (1976). "Reasons of explosive spalling of concrete by fire." Beton I zhelezobeton (Concrete and Reinforcement Concrete)(3).

APPENDIX A EFFECT OF VARIOUS POLYMER FIBERS ON PREVENTING SPALLING

A.1 Mix proportion and experiments

To study the effect of various fibers on spalling prevention of UHPC, linear low-density polyethylene (LLDPE), ultra-high molecular weight polyethylene (UHMWPE), polyamide 66 (PA66) and polyester (PET) fibers were used in the mix mentioned in CHAPTER 3 and the samples were denoted as UHPC-LLDPE, UHPC-UHMWPE, UHPC-PA66, and UHPC-PET, respectively. A dosage of 3 kg/m³ of fibers was added into each mix. For all the fibers, the diameter and length were 30 μ m and 12 mm, respectively. The mix proportion is shown in Table A.1. Permeability and spalling tests were conducted. DSC, TMA, and SEM were carried out following the same procedures as mentioned in CHAPTER 3.

Table A.1. Mixture compositions of UHPC.

Mix design	W/B	C	SF	SS	SP	FA	W	Fibre	f_c (MPa)
UHPC-LLDPE	0.2	1.0	0.25	0.25	1.1	0.04	0.25	3	140.6
UHPC-UHMWPE	0.2	1.0	0.25	0.25	1.1	0.04	0.25	3	137.8
UHPC-PA66	0.2	1.0	0.25	0.25	1.1	0.04	0.25	3	137.91
UHPC-PET	0.2	1.0	0.25	0.25	1.1	0.04	0.25	3	133.9
W/B: water binder ratio					SS: Silica sand				
C: cement					SP: superplasticizer				
SF: silica fume					FA: fine aggregate.				
f_c : compressive strength at 28 days									

A.2 Results and discussion

Fig. A.1. Spalling test results: (a) UHPC-LLDPE, (b) UHPC-UHMWPE, (c) UHPC-PA66 and (d) UHPC-PET.

Fig. A.1 shows the spalling test results of UHPC with different polymer fibers. As evident, not all of the polymer fibers could prevent spalling of UHPC. Both UHPC-UHMWPE and UHPC-PET showed severe spalling after heating, while UHPC with LLDPE and PA66 fibers showed good spalling resistance. From DSC results in Fig. A.2, it was found that the melting point of LLDPE, UHMWPE, PA66, and PET fibers were 123.15 °C, 143.62 °C, 258.66 °C, and 255.43 °C, respectively. It was extremely interesting to find that UHMWPE fibers with a lower melting point (~143 °C) than PP fibers did not help prevent spalling of UHPC, while LLDPE fibers, which also has a lower melting point than PP fibers, could prevent spalling of UHPC. Meanwhile, although PA66 and PET fibers had a similar melting point (~255 °C), their effectiveness on spalling prevention of UHPC was different. Despite a higher melting

point than PP fibers, PA66 fibers could enhance spalling resistance of UHPC considerably. In contrast, PET fibers were not effective for spalling prevention. The results presented here clearly showed that the melting point of polymer fibers was not a dominant parameter in preventing spalling.

Fig. A.2. DSC results of different polymer fibers.

Fig. A.3. Thermal strain of polymer fibers with increasing temperature.

Table A.2. Melting point and CTE of polymer fibers.

Polymer fibers	Melting point (°C)	CTE ($10^{-3}/^{\circ}\text{C}$)*
LLDPE	123.15	1.4 (25~105 °C)
UHMWPE	143.62	0.2 (25~140 °C)
PP	169.58	1.0 (25~150 °C)
PA66	258.66	0.7 (25~250 °C)
PET	255.43	0.6 (25~250 °C)

*CTE is measured before melting of fiber. The testing temperature is remarked for each fiber.

Fig. A.4. SEM images of UHPC with polymer fibers: (a) UHPC-LLDPE after exposure to 105 °C, (b) UHPC-UHMWPE after exposure to 105 °C, (c) UHPC-PA66 after exposure to 200 °C, and (d) UHPC-PET after exposure to 200 °C.

In fact, as mentioned in CHAPTER 3, thermal mismatch between polymer fibers and cement matrix is considered as a key factor for preventing spalling. The TMA data in Fig. A.3 clearly shows the thermal expansion of different polymer fibers. Most of the fibers, except UHMWPE fibers, showed very large thermal expansion before melting. Although the thermal strain of UHMWPE fibers was larger than cement paste at high temperature, it was much smaller than those of the other fibers. The maximum thermal strain of UHMWPE was only 1/5 to 1/8 of the thermal strain of other fibers. In addition, it was also found that although PA66 and PET fibers had a similar melting point, thermal expansion of PET fibers was much smaller than that of PA66 fibers.

SEM micrographs showing the effect of thermal expansion of polymer fibers on microstructure of UHPC samples are presented in Fig. A.4. The high thermal expansion of LLDPE, PA66 and PET fibers could create microcracks around fibers before they started melting, as shown in Fig. A.4. As LLDPE fibers expanded significantly above 60 °C, microcracks were formed before 105 °C, resulting in a high permeability value of UHPC-LLDPE at this temperature (Fig. A.5). Consequently, spalling of UHPC was prevented. Similarly, permeability of UHPC-PA66 and UHPC-PET increased significantly at 200 °C (Fig. A.5), due to formation of microcracks (Fig. A.4c and d). But due to lower thermal expansion of PET fibers, permeability of UHPC-PET was lower than that of UHPC-PA66. Therefore, the effectiveness of PET fibers on preventing spalling was worse than that of UHPC-PA66. In contrast, only limited fine microcracks were found around UHMWPE fibers after exposure to 150 °C. As a result, the permeability of UHPC-UHMWPE did not see any significant increase with increasing temperature. Thus, spalling still occurred in UHPC with UHMWPE fibers (Fig. A.1b).

In addition, Fig. A.6 presents the SEM images of UHPC-LLDPE and UHPC-UHMWPE fibers after exposure to 200 °C. It clearly showed that molten polymer

fibers flowed out of the initial channels and remained on the surface of samples. Absorption of molten fibers did not happen in UHPC. The permeability and spalling test results (Fig. A.1 and Fig. A.5) indicated that the absorption of molten fibers (which resulted in empty channels) was not necessary for preventing spalling. Clearly, the findings here reaffirm the importance of thermal mismatch on preventing spalling of UHPC.

Fig. A.5. Hot permeability of UHPC with different polymer fibers.

Fig. A.6. SEM micrographs of (a) UHPC-LLDPE and (b) UHPC-UHMWPE after exposure to 200 °C.