Diffusion between glass and metals for optical fiber preform extrusion

Felicia Yan Xin Yeo, Zhifeng Zhang, Dileep Kumar Chakkathara Janardhanan Nair, Yilei Zhang*
School of Mechanical and Aerospace Engineering, Nanyang Technological University, 50 Nanyang
Avenue, Singapore 639798

ABSTRACT

When silica is extruded, diffusion of metal atoms into silica results contamination to the silica being heated, and thus is a serious concern for the glass extrusion process, such as extrusion of glass fiber preform. This paper examines diffusion between fused silica and two high strength metals, the stainless steel SS410 and the superalloy Inconel 718, at 1000 °C and under the normal atmosphere condition by SEM and Electron Dispersion Spectrum. It is found that diffusion occurs between silica and SS410, and at the same time, SS410 is severely oxidized during diffusion experiment. On the contrary, the diffusion between Inconel 718 and silica is unnoticeable, suggesting excellent high temperature performance of Inconel 718 for glass extrusion.

Keywords: glass extrusion, glass-metal diffusion, optical fiber preform

1. INTRODUCTION

Diffusion between metals and ceramics is of fundamental technical importance in various applications and has been studied for several decades [1,2]. For example, silicon nitride, used as tool material for machining iron, wear rapidly due to the diffusion process [3]. It has been found previously that when silica is heated in metal tools, diffusion of metal atoms into silica contaminate silica sample while metal tools wear more rapidly due to the presence of the silica [10]. Another area involves this diffusion process is the integrated circuit, in which metal conductors make contact with dielectric silica. Diffusion of metal atoms into silica or silicon wafer substrate has detrimental effect on the whole circuit and thus has been closely investigated. It has been found that various metal elements could diffuse into silica though the quantity varies [4–7,2]. These studies investigate elemental metals like Cu, Ag etc used in the semiconductor industry, and mainly examine the influence on electrical properties.

This paper is particularly focused on the diffusion between silica and two high-temperature alloys, which is one of several issues facing the development of the extrusion system for silica preform fabrication. Figure 1 illustrates the concept of glass preform extrusion. Melt silica or other glasses is extruded through the die to form preform for drawing optical fiber. Fabrication of structured preform for drawing microstructured optical fiber by extrusion has been realized for soft glass, and proved to be a versatile technique [8]. To apply the method to silica, however, is still a technical challenge because of the high temperature involved (about >1700 °C). One of the major difficulties is to find a strong enough material for extrusion tools that can withstand such high temperature and at the same time does not contaminate the silica. Contamination of the silica will inevitably results in optical fibers with high loss, which is adverse for optical fiber applications [9]. The SS410 and the Inconel 718 are two of common alloys for high temperature applications [11,12]; but their diffusion with silica under temperature around 1000°C has not been studied, which is the focus of this paper.

---

* ylzhang@ntu.edu.sg

International Conference on Optical and Photonic Engineering (icOPEN 2015), edited by Anand K. Asundi, Yu Fu,
Proc. of SPIE Vol. 9524, 95242S · © 2015 SPIE · CCC code: 0277-786X/15/$18 · doi: 10.1117/12.2190640

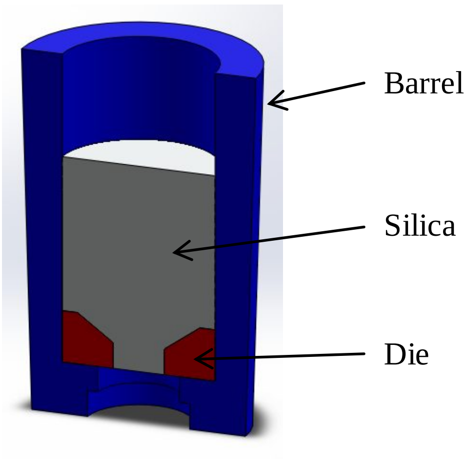

Figure 1 Diagram of extrusion of silica preform for drawing optical fiber

2. EXPERIMENTS

The experimental procedures are shown in

Figure 2. Slices of fused silica, SS 410 and Inconel 718 are cut from long rods and their surfaces are polished by silicon carbide papers with grid size from P#200 (Struers) initially to P#2400 finally. For diffusion experiment, a silica slice is placed on a SS410 or Inconel 718 sample and then heated in the furnace under the normal atmosphere condition. The temperature was slowly increased to 1000 °C at a speed of 2 °C/min, and maintained at 1000 °C for six hours, and then decreased to room temperature at a speed of 1 °C/min. After heat treatment the surfaces making contact were characterized by SEM (JEOL JSM-5600LV) and Electron Dispersion Spectrum (EDS). A gold coating is coated on silica surfaces for SEM observation.

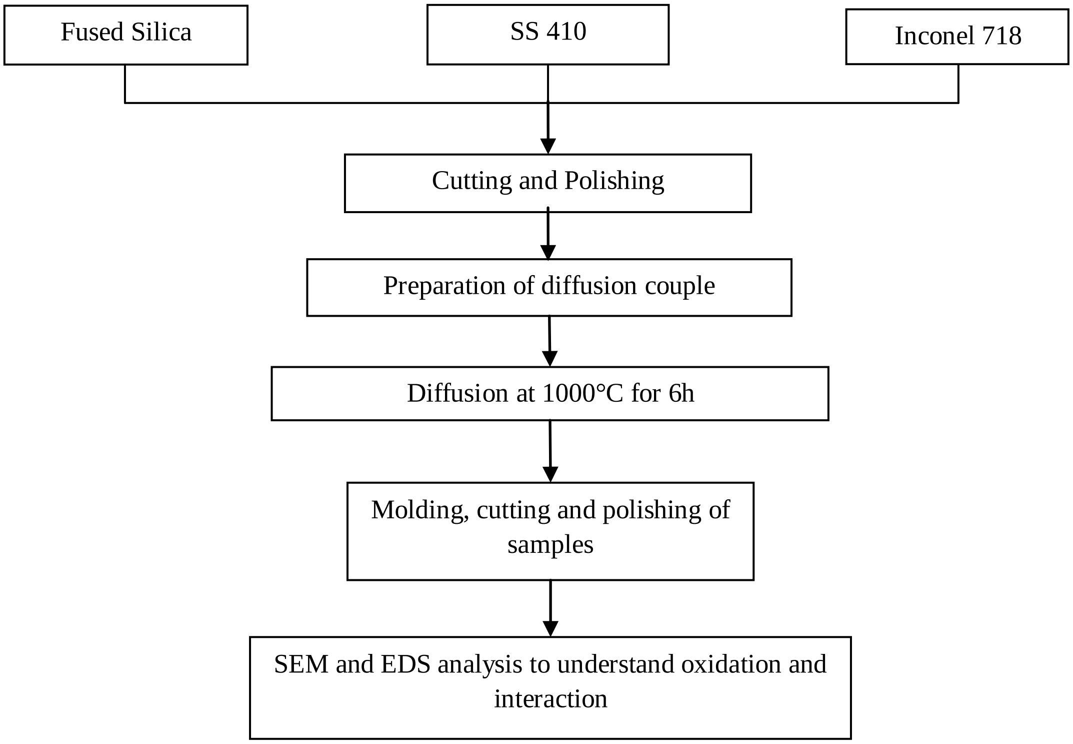

Figure 2 Diagram of the experimental procedures

3. RESULTS & DISCUSSIONS

Figure 3 shows the fused silica slices on SS410 and Inconel 718 samples after heat treatment. Evidently severe oxidation and spalling of SS410 occurred. In contrast, there is no detectable contamination on the fused silica surface on Inconel 718 and the fused silica remains clear with no coloration. Since nothing is sticking onto the fused silica sample, it can be safely concluded that there is no reaction between fused silica and Inconel 718.

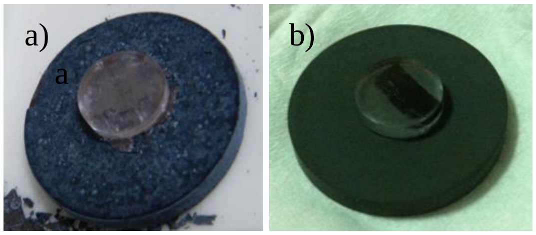

Figure 3 Photos of diffusion couple of silica on a) SS410 and b) Inconel718 after heat treatment

Figure 4 shows the SEM image of the silica on SS410 sample after heat treatment.
Elemental analysis was measured at nine locations on the fused silica surface on the SS 410 sample after diffusion, as shown in Figure 5. Through the EDS measurement, different elements that are present in the selected spot or area can then be determined. Apart from showing the different elements, the amount of each element in atomic percentage is also measured. Additionally, the result provides information on the mean, standard deviation, maximum and minimum amount of each element across the spectrums as well. The element Fe has presence at al positions. Due to spalling of scale from SS410 sample, SEM and EDS analysis was not performed on SS410 sample.

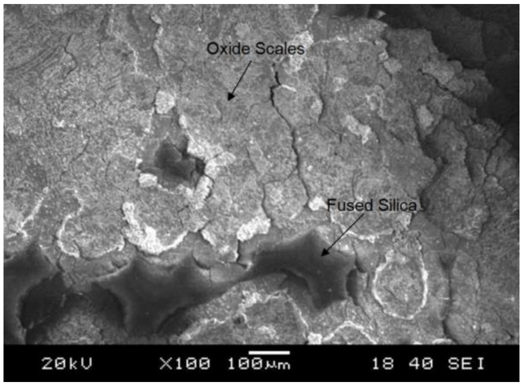

Figure 4 SEM image of the silica surface on SS410 after heat treatment. The oxide scales come from the oxidation of SS410 sample when it is in the furnace.

For the spectrum 1 and the according position, it consists of 98.04% Si, 0.98% Fe and 0.98% Nb, suggesting that that the fused silica part has a bit of contamination from iron and niobium. And this explains why the fused silica sample has a reddish coloration. The Spectrum 5, which locates at the boundary of the oxide scale and the fused silica, consists of only 5.43% of silicon. The main element here is iron which has an atomic percentage of 92.55%. The rest of the elements that made up a small percentage are Cr (Chromium), Mn (Manganese), Co (Cobalt) and Nb (Niobium) respectively. Spectrum 9 is located right on the oxide scale itself. The main element there is Fe with an atomic percentage of 97.00%.

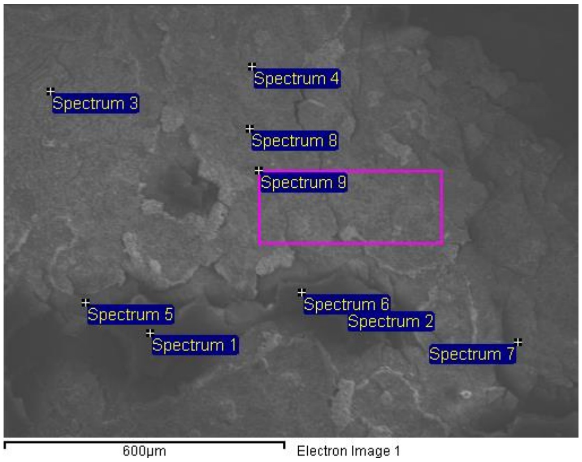

Figure 5 Nine positions for EDS measurement of on the silica surface

Figure 6 shows the EDS result of the fused silica sample before heat treatment, and only Oxygen with an atomic percentage of 72.17% and with an atomic percentage of 27.83% are detected. This means that there is no contamination beforehand. The fused silica sample becomes contaminated only after the heat treatment process.

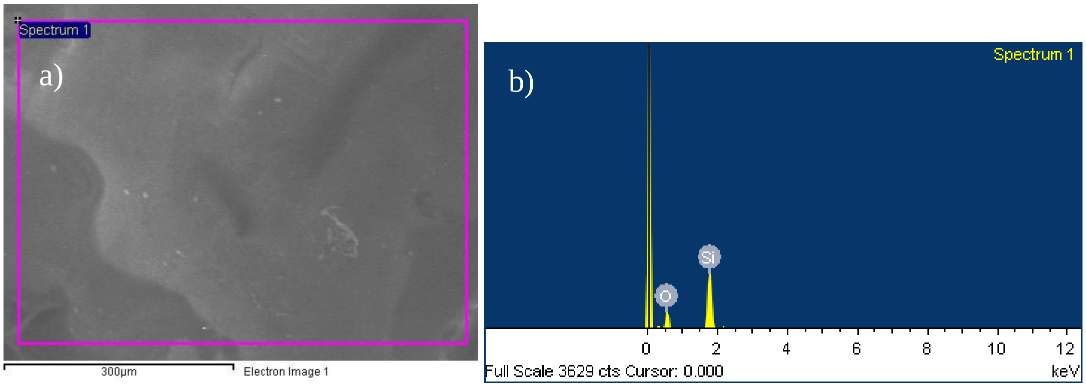

Figure 6 EDS result of the fused silica before diffusion experiment a) SEM b) EDS

Figure 7a is the SEM image of the Inconel 718 surface making contact with fused silica after heat treatment. The secondary structure of nodules in spherical or irregularly rounded are observed. Figure 7b is the SEM image of the fused silica on Inconel 718.

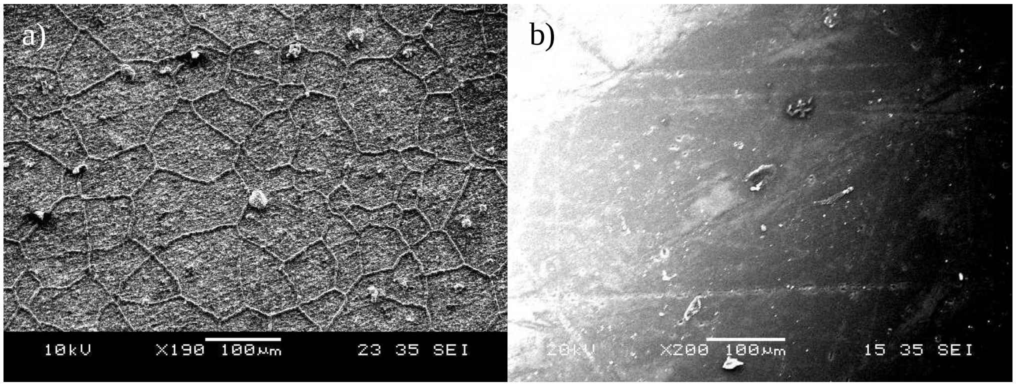

Figure 7 a) SEM picture of the surface of Inconel 718 sample after heat treatment; b) SEM image of the fused silica surface making contact with Inconel 718 after heat treatment

Figure 8a is the result of EDS of the surface of the Inconel 718 sample after diffusion treatment. It shows that the main element is Cr which has an average atomic percentage of 75.48%. There is also presence of Ti, Ni, Fe and Nb whereby the atomic percentage is 19.42%, 0.36%, 2.31% and 0.83% respectively. The result also suggests that there is no silicon diffusion into the Inconel 718 sample as there is 0% of silicon present in the Spectrum. This is favorable as contamination is unwanted. However, the measured composition of Inconel 718 surface after diffusion experiment considerably differs from its reference composition (Figure 7b).

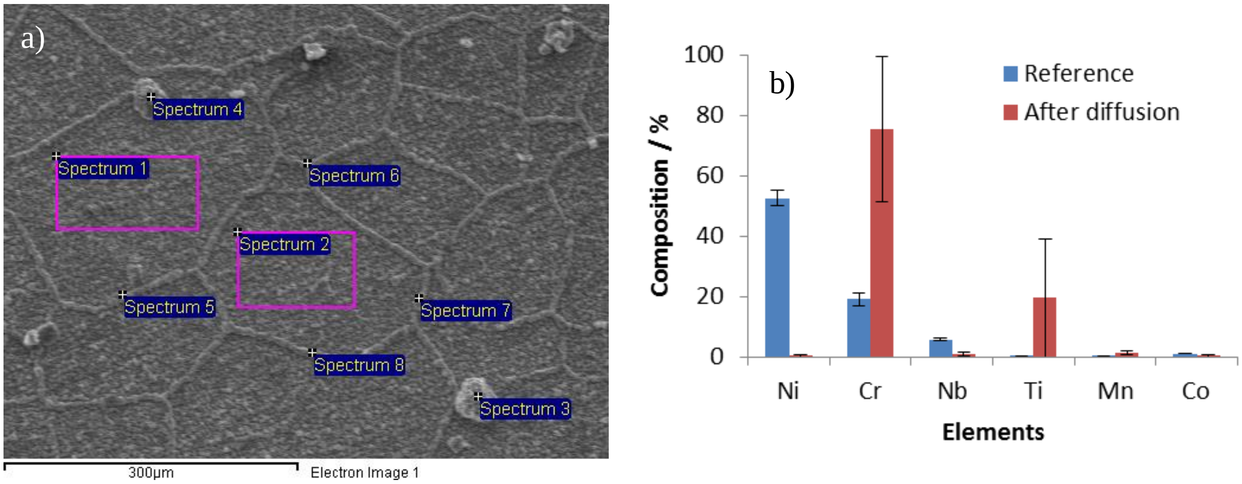

Figure 8 a) Nine positions for EDS measurement of on the Inconel 718 surface; b) Comparison of composition of Inconel 718 before (reference) and after diffusion treatment

The atomic percentages of O and Si of the silica surface after heat treatment are measured to be 63.09% and 36.91%, as shown in Figure 9. It further confirms that there is no contamination or any other reaction between Inconel 718 and fused silica.

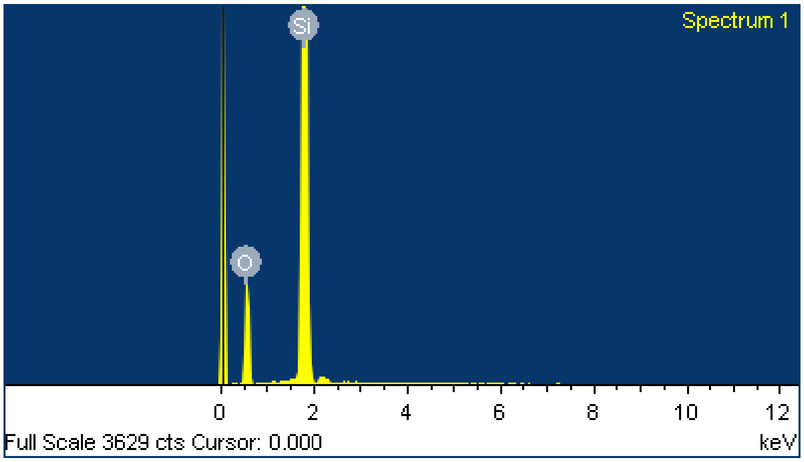

Figure 9 EDS result of the silica surface after diffusion on the Inconel 718 sample

4. CONCLUSION

Diffusion couples of silica and two metals are studied by SEM and EDS measurements for glass extrusion process. The diffusion between stainless steel SS410 and silica is evident and EDS results reveals metal elements on surface of silica. Additionally, SS410 is severely oxidized and degraded due to heat treatment in the presence of oxygen. In comparison, no diffusion between silica and superalloy Inconel 718 is detected, suggesting excellent thermal stability of Inconel 718 and making it potential candidate for glass extrusion under high temperature.

REFERENCES

[1] R.N. Ghoshtagore, Diffusion of Nickel in Amorphous Silicon Dioxide and Silicon Nitride Films, J. Appl. Phys. 40 (1969) 4374. doi:10.1063/1.1657201.
[2] M. He, T.-M. Lu, Metal-Dielectric Interfaces in Gigascale Electronics, 157 (2012). doi:10.1007/978-1-4614-1812-2.
[3] F.J. Oliveira, R.F. Silva, J.M. Vieira, Interfacial Reaction Kinetics of Silicon Nitride/Iron Alloys Diffusion Couples in the Range 1050° C/1250° C, in: Interfacial Sci. Ceram. Join., Springer, 1998: pp. 203–209.
[4] K. Hozawa, J. Yugami, Copper diffusion behavior in SiO2/Si structure during 400° C annealing, Jpn. J. Appl. Phys. 43 (2004) 1.
[5] K. Hozawa, S. Isomae, J. Yugami, Copper distribution near a SiO2/Si interface under low-temperature annealing, Jpn. J. Appl. Phys. 41 (2002) 5887.
[6] D.A. Ramappa, W.B. Henley, Diffusion of Iron in Silicon Dioxide, 146 (1999) 3773–3777.
[7] H.G. Francois-Saint-Cyr, F. a. Stevie, J.M. McKinley, K. Elshot, L. Chow, K. a. Richardson, Diffusion of 18 elements implanted into thermally grown SiO[sub 2], J. Appl. Phys. 94 (2003) 7433. doi:10.1063/1.1624487.
[8] V.V.R. Kumar, a George, W. Reeves, J. Knight, P. Russell, F. Omenetto, et al., Extruded soft glass photonic crystal fiber for ultrabroad supercontinuum generation., Opt. Express. 10 (2002) 1520–5.
[9] P. Kaiser, Contamination of furnace-drawn silica fibers, Appl. Opt. 16 (1977) 701–704.
[10] H. Dallaporta, M. Liehr, J.E. Lewis, Silicon dioxide defects induced by metal impurities, Phys. Rev. B. 41 (1990) 5075.
[11] M.F. McGuire, Stainless steels for design engineers, ASM International, Materials Park, Ohio, 2008.
[12] M.J. Donachie, S.J. Donachie, Superalloys a technical guide, ASM International, Materials Park, OH, 2002.