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# Characterization of Low Pressure RF Plasma Heating

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**Abstract:** Compacts of α-$Al_2O_3$ and Mo powder were heated in radio-frequency (RF) induced low pressure $N_2$, $H_2$, Ar, and their mixture plasma. An optical pyrometer, a radiation pyrometer, and a system called Accufiber Model 310 were used to measure the temperature of compacts heated in the plasma. The results indicate that there are different heat transfer mechanisms from plasma to specimens of different physical properties. The Ar plasma showed the highest heating ability among $N_2$, $H_2$, and Ar plasma for $Al_2O_3$ specimens, whereas the $H_2$ plasma could heat Mo specimens to a higher temperature than the Ar plasma did, even under the same generating conditions.

**Key words:** plasma heating; temperature measurement; heat transfer mechanism

The heat transfer process from plasma to specimens is much more complicated than in the case that specimens are heated in an ordinary high temperature atmosphere. Some experimental investigation and theoretical analysis of plasma-wall heat transfer have been reported[1-4]. The existence of free electrons, ions, and neutral species in a plasma and the steep gradients of the plasma parameters, particularly in the vicinity of walls, give rise to a number of peculiar effects which are still poorly understood. Generally, examination of a heat transfer process is based on the measurement of heating procedure. However, the accurate measurement of specimen temperature heated in plasma is very difficult. For examples, some significant errors could be introduced into an optical pyrometric measurement, because of the interference from the luminous plasma and the uncertain emissivity of the specimen surface; a RF plasma operation causes a strong electric noise which will interfere the measurement using a bimetallic thermocouple element.

In this study, a variety of methods for temperature measurement was used, such as by an optical and a radiation pyrometers, an Accufiber system, to measure the temperature of $Al_2O_3$ and Mo compacts of different physical properties, and to give a comprehensive examination on the heating characteristics of low pressure high intensity RF plasma.

## 1 Experiment

A schematic drawing of the torch part of plasma heating apparatus is shown in Fig. 1. High-purity $N_2$, $H_2$, Ar, and their mixtures were supplied through a gas controlling system into the plasma torch which consists of a water-cooled quartz tube and an induction coil. A 5 MHz power supply capable of 15 kW plate power was used to generate the plasma. Samples can be set at different positions along the axis of the tube by a boron nitride holder. The pressure of the plasma working gas was fixed at 80 Pa.

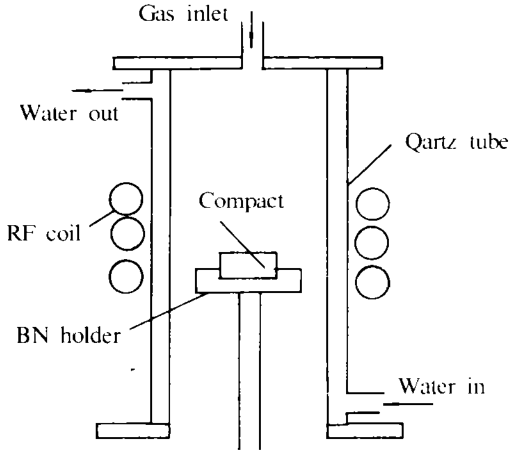

**Fig. 1 Schematic view of the plasma heating apparatus**

$Al_2O_3$ and Mo powder compacts of 3 μm grain size, with green densities of about 50% and 68% of their theoretical value respectively, were heated in the plasma and their temperatures were measured by three kinds of methods. The first was to use an optical pyrometer of disappearing filament to measure the equilibrium temperature of the specimens. Errors caused by the luminous plasma were evaluated, by comparing the measured equilibrium temperature in plasma with the value obtained by extrapolating the cooling curve measured after quenching the plasma. The second was to measure the time variation of specimen temperature, using a radiation pyrometer which can respond to the quick change of the specimen temperature. The third was to use a system called Accufiber Model 310. Fig. 2 shows the schematic drawing of its probe part. This system has a sapphire fiber coated with thin film of platinum alloy and sheathed with a sapphire tube, forming a probe about 3 mm in diameter. The probe can be set into the torch and heated directly by the plasma. Signals from the coated film are conducted by the fiber to a detector where they are transformed into value of temperature.

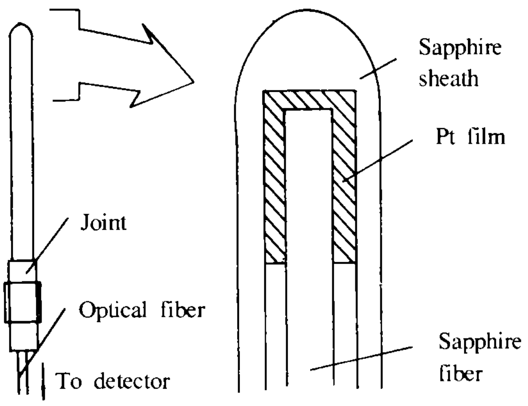

**Fig. 2 Schematic view of the probe part of the temperature measurement system called Accufiber Model 310**

## 2 Results and Discussions

### 2.1 Heating of $Al_2O_3$ specimens

Table 1 shows temperatures of $Al_2O_3$ compacts heated in plasmas for 120 s measured with the optical pyrometer, and the equilibrium temperatures of the Accufiber probe. Two things can be noticed from this table. At first, temperatures of the probe rise with the increase of the plate power. Secondly, the probe temperatures reveal that the $H_2$ plasma exhibited a higher heating ability than the $N_2$ plasma generated at the same plate power of 6 kW. This could be due to the fact that $H_2$ has a lower dissociation energy and a higher thermal conductivity than that in the $N_2$ case. However, the compact heated in the $N_2$ plasma exhibited a temperature of 1 650℃ which is 100℃ higher than the value of 1550℃ reached in the $H_2$ plasma, which shows inconsistency with the probe measuring.

**Table 1 Temperature of $Al_2O_3$ compacts* heated for 120s and equilibrium temperature of sapphire probe****

| Method | Plasma gas | Plate power / kW | Temperature / ℃ |
|---|---|---|---|
| compacts | $N_2$ | 6 | 1 650 |
|  | $H_2$ | 6 | 1 550 |
| sapphire probe | $N_2$ | 4.5,6,8 | 1 200,1 390,1 560 |
|  | $H_2$ | 6 | 1 480 |

Note: * by optical pyrometer and the data were compensated with $\varepsilon = 0.3$;

** by Accufiber Model 310, with the top of the probe set at the same position as the top surface of the compacts

Fig. 3 shows the time—temperature dependence of the $Al_2O_3$ compacts measured with the radiation pyrometer. Temperatures passed through a maximum either heated in the $N_2$ or in the $H_2$ plasma. But the curves show different "cool-down" characteristics. This indicates that compacts heated in the $N_2$ and $H_2$ plasma will exhibit different relative temperature values with the change of time. The maximum temperature of the compact heated in the $H_2$ plasma is higher than that heated in the $N_2$ plasma. However, at 120 s corresponding to the measured values show in Table 1, the compact heated in the $N_2$ plasma revealed a temperature higher than that heated in the $H_2$ plasma. This just clarified the inconsistency of the results shown in Table 1.

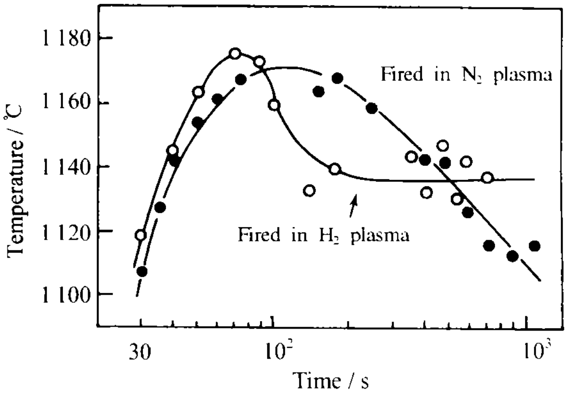

**Fig. 3 Time—temperature dependence of $Al_2O_3$ compacts measured with the radiation pyrometer**

E L Kemer, et al.[5] reported that the cool-down phenomenon occurred only in the case of Ar plasma heating but not in $N_2$, $H_2$ or other diatomic gas plasma heating. It was found in the present study that not only Ar but also $N_2$ or $H_2$ plasma heating of $Al_2O_3$ compacts exhibited the cool-down effect, with some difference in character when the plasma gas or the specimen conditions was changed. The time—temperature dependence in Fig. 3 corresponds to different densification behavior shown in Fig. 4. That is, the turning points of the curves in Fig. 3 correspond to the ones of densification rates in Fig. 4, at about 75 s in the case of $H_2$ plasma heating and at 110 s in the $N_2$ case of $N_2$. This behavior could be attributed to the change of heat transfer rate from plasma to compact resulting from the decreasing porosity in compact during sintering[3].

Among $H_2$, $N_2$, and Ar plasma, the Ar plasma showed the highest heating ability for $Al_2O_3$ compacts. It could even melt the compact surface and the sapphire probe of the Accufiber at a plate power of 8 kW, whereas the $H_2$ or $N_2$ plasma could not heat the specimens to that extent at the same plate power. The specimen densified most rapidly obtaining a density of 82% of the theoretical value in the Ar plasma at a plate power of 4.5 kW, compared with those fired in the $N_2$ and $H_2$ plasma at a plate power of 6 kW, see Fig. 5. Meanwhile, the specimen surface in Fig. 5(C) fired in the Ar plasma seems to reveal partial melted trace and shows relatively smooth morphology than those fired in the $N_2$ and $H_2$ plasma. The different surface morphology in Fig. 5 shows the different interaction effects of the compacts with the three kinds of plasmas.

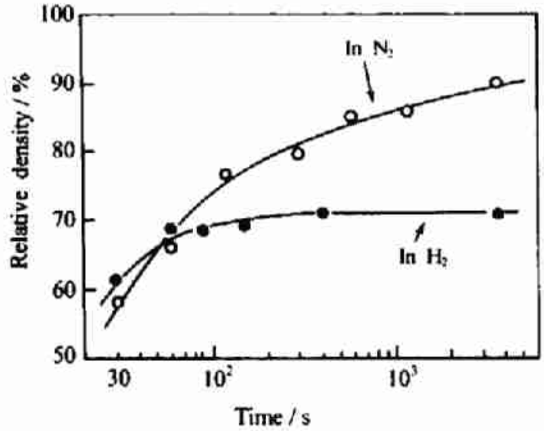

**Fig. 4 Relation between relative density of $Al_2O_3$ compacts and firing time in plasma of 6kW**

### 2.2 Heating of Mo specimens

Table 2 shows the measured temperatures of Mo specimens. The $H_2$ plasma revealed higher heating ability than Ar plasma for Mo specimens, which is contrary to the heating of $Al_2O_3$ specimens in the plasma of these two gases. Furthermore, we can notice from Table 2 that the temperature of the Mo compact heated in Ar + 50% $H_2$ plasma was higher than that heated in any plasma of its component gas at 8 kW.

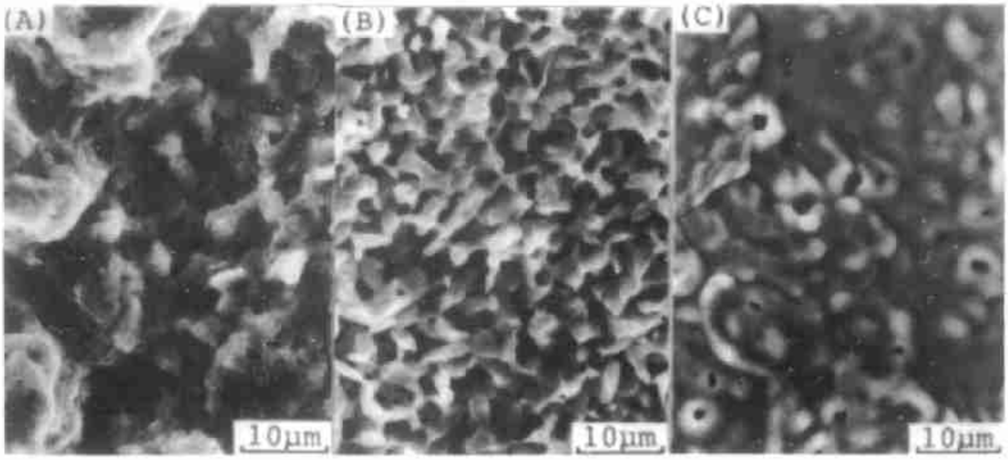

**Fig. 5 Surface morphologies of $Al_2O_3$ compacts fired (A) in $N_2$ plasma of 6kW for 120s obtaining a relative density of 76%, (B) in $H_2$ of 6kW for 400s obtaining 71%, and (C) in Ar of 4.5kW for 120s obtaining 82%**

**Table 2 Temperature* of Mo compacts measured with the optical pyrometer**

| Gas type | Temperature / ℃ | |
|---|---|---|
| | at 8 kW | at 10 kW |
| $H_2$ | 1 730 | 1 900 |
| Ar+50% $H_2$ | 1 820 | 1 900 |
| Ar | 1 610 | 1 700 |

Nate: * as measured without compensation

Fig. 6 shows the density of Mo compacts fired in Ar, $H_2$, and their mixtures plasma at 10 kW for 180 s. A high heating ability of plasma generally causes rapid densification at the early stage of sintering, although it does not always result in good effect in the whole sintering procedure. Thus, the results in Fig. 6 appear to be consistent with those in Table 2. This could be attributed to that $H_2$ has a very low dissociation energy

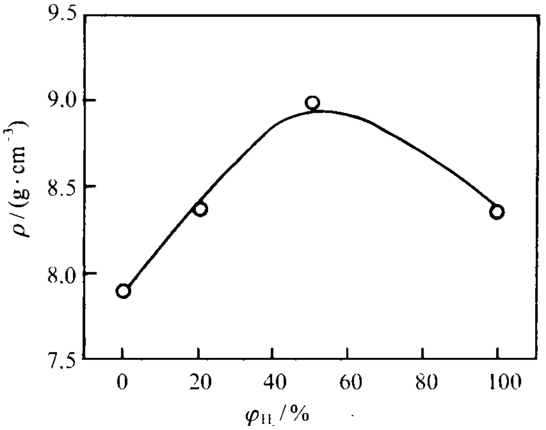

**Fig.6 Relation between sintered density of Mo compact and $H_2$ content of Ar+$H_2$ plasma at 10kW fired for 180s**

(4.48 eV) and its thermal conductivity is much higher than Ar gas in the range up to $2\times10^4$ K[6]. The composite effect of the high gas temperature of Ar[7] and the high thermal conductivity of $H_2$ caused the high heating ability of the Ar and $H_2$ mixture plasma.

### 2.3 Problems of temperature measurements

The results on the temperature measurement of $Al_2O_3$ compacts showed that the luminosity of plasma caused an error of 30～50℃ higher than the actual temperature of the specimens, when the optical pyrometer was used. The morphology change of specimen surface, according to the different plasma-wall interaction and densification conditions, causes difficulty to estimate the real emissivity of the specimen, which also introduces errors to temperature measurement by the optical pyrometer.

The data in Fig. 3 show that the temperature measured with the radiation pyrometer appears to be about 400℃ lower than that measured with the optical pyrometer (see Table 1). The temperature measurements with the optical and radiation pyrometers act in the wavelength range of 650～1 100 nm. The quartz tube and the cooling water of the plasma torch do not show a significant absorption in this range of wavelength. However, the working mechanism of these two pyrometers is different. In the case that the optical pyrometer is used, it only needs to compare the color of the specimen with that of the filament. And the radiation pyrometer measures the emission energy from a certain area of the specimen. The refraction at the interface of the quartz tube and cooling water might affect the temperature measurement and cause errors, when the radiation pyrometer is used.

The results measured by the Accufiber Model 310 correctly reflected the effects of gas species on the heating characteristics of plasma for $Al_2O_3$ compacts, but they cannot explain why the plasma heating of Mo compacts is different from that of $Al_2O_3$ compacts. This could be interpreted to be that the probe of the Accufiber system is sheathed by sapphire which is the same material as $Al_2O_3$.

### 2.4 Discussions on heating mechanism

To interpret the different heating characteristics for Mo and $Al_2O_3$ specimens, some discussions will be given based on the present results and theoretical analysis in other reports.

E Pfender, et al. [3] analyzed the heat transfer of plasma sintering process taking the case of $Al_2O_3$ as an example. They revealed that heat transfer from plasma to the wall depends strongly on catalytic properties of the surface which results in different boundary layer separating free stream of plasma from the wall. In the case of non-catalytic surface, negative charge on the surface gives rise to an electric field which increases the rate and energy of the ions arriving at the surface, resulting in a substantial increase of the heat transfer. That is, the ion density in plasma greatly affects the heat transfer rate from plasma to specimen of electric insulator. The density of ions in an Ar plasma is generally higher than that in a $H_2$ or $N_2$ plasma at the same generating conditions, which result in high heat transfer from Ar plasma to $Al_2O_3$ compacts. This just interprets the results on the heating of $Al_2O_3$ specimens in the present work.

On the other hand, G R Chludzinski[2] determined the heat transfer rate of Ar and Ar + $N_2$ plasma, by measuring the $dT/dt$ with a inconel-sheathed chromel-alumel thermocouple and analyzing the measured results. His results showed that reactions of the atomic recombination are more important than the ion-electron reactions. This suggests that recombination reactions of dissociated atoms in diatomic gas plasma play an important role in enhancing the heat transfer to a specimen of good conductivity, such as inconel in Chludzinski's work and Mo in this study.

Accordingly, E Pfender, et al. and G R Chludzinski analyzed the plasma heating processes based on the temperature measurements of samples of different physical properties. A comprehensive review of the available information on experiment and analysis indicates that we should consider that there are different heat tran-s-fer mechanisms from plasma to specimen, when the physical properties of heated specimens are different even under the same plasma conditions.

## 3 Conclusions

The heating characteristics of the low pressure RF plasma for specimens of different physical properties are quite different. The Ar plasma heated $Al_2O_3$ specimens most effectively among the $N_2$, $H_2$, and Ar plasma, whereas the $H_2$ plasma was more capable to raise the temperature of Mo specimens than the Ar plasma. There could be two mechanisms which dominate the heat transfer rate from plasma to specimen: ion density in plasma greatly affects the heating characteristics when the specimen is an electrical insulator; thermal conductivity, especially the portion caused by the recombination of dissociated atoms of plasma gas plays an important role in heating the specimens of good electrical conductivity.

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