Interfacial Reaction between Zirconium Alloy and Zirconia Ceramic Shell Mold

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Abstract: The ceramic shells were fabricated with ceramic slurry containing zirconia as filler material and colloidal silica binder to cast Zr-alloy 705. The investment shell mould characteristics in terms of hot bending strength, thermal expansion and metal-mould reaction were measured. On account of phase transformation (confirmed by Raman spectra) of zirconia at high temperatures the hot bending strength zirconia shells decreases with increasing temperature. A hard ZrO_2 case layer was noticed beneath the surface of the casting.

Keywords: Investment shells, zirconia, hot strength, thermal expansion, metal-mould reaction.

1. Introduction

Zirconium alloys have a wide range of applications in fields such as atomic energy, chemical industry, light industry, and machinery [1]. Most of zirconium alloy products are manufactured by forging technology. This is mainly due to the fact that the casting technology of zirconium is unstable and not yet well established, especially in selecting the mold materials, which are difficult for users to determine. The materials used to build the investment shell moulds, especially refractories, play a vital role in the production of quality castings [2-7]. The properties of refractory fillers, which affect the shell quality, are melting point, thermal expansion, and metal - mould interaction. During the casting process, molten zirconium alloys can easily react with the mold materials and produce a surface contamination layer.

In the present work, zirconia was used as refractory filler material to fabricate investment shell moulds for casting of zirconium alloys.

2. Materials and Methods

The colloidal silica binder was used to fabricate the ceramic shells from zirconia as a reinforced filler material. Three phases of zirconia are monoclinic <1170 °C, tetragonal 1170–2370 °C, and cubic >2370 °C (figure 1). The specifications of colloidal silica binder and zirconia are, respectively, given in table 1 and table 2. Two grades (primary and backup sands) of stuccoing sand were employed in the present investigation.

Primary sand: A finer grade silica sand having AFS grain fineness number 120. This is a synthetic sand. This sand was used for first two coats, called prime coats to get good surface finish and every detail of the wax pattern.

Backup sand: A coarser grade sand having AFS grain fineness number 42. This sand was used for the rest of coats, called backup coats on the ceramic shells. This is a river sand.

The backup sand was employed to develop more thickness to the shell walls with minimum coats.

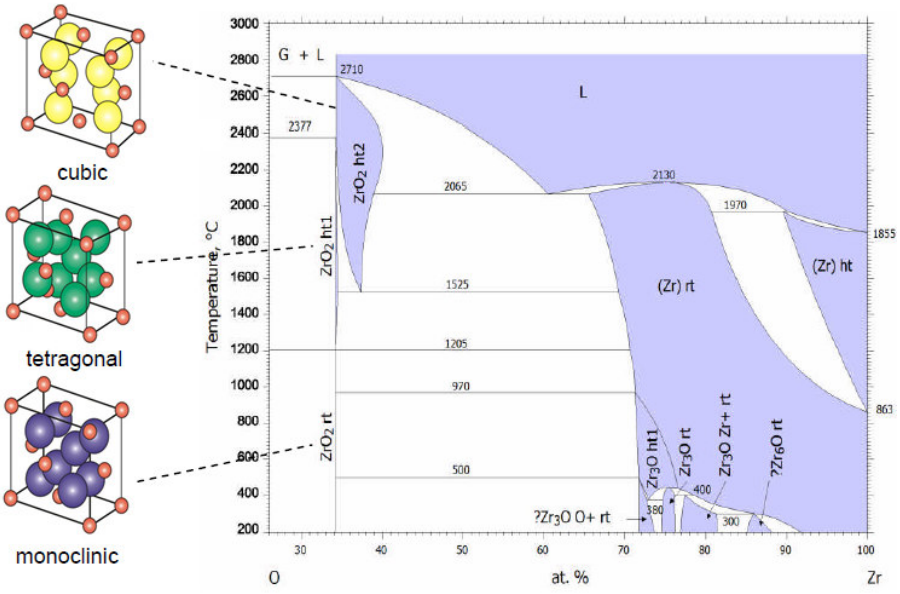

Figure 1. Crystal structure of zirconia

Table 1. Specifications of silox binder

Property	Amount
Silica (SiO_2) Wt%	30
P^H at 25^0C	10.5
Titrable Alkali (Na_2O)	0.6
Chlorides/ Sulphates	Traces
Specific gravity, g/cc	1.23

Table 2. Specifications of zirconia

Property	Zirconia
Density, g/cc	5.68
Refractoriness, ^0C	2715
Chemical composition	ZrO_2
Sieve analysis	200-mesh (74 μm)

2.1 Manufacture of ceramic shells and Zr-alloy 705 castings

The investment shells were made of applying a series of ceramic coatings to the wax patterns. The pattern was first dipped into the dip-coating slurry bath. The pattern drains off excess slurry and to produce a uniform layer. The wet layer was immediately stuccoed with coarse silica sand. Each coating was allowed to dry in the open air. The operations of coating, stuccoing, and drying were repeated six times. The seventh coat was left unstuccoed to avoid the occurrence of loose particles on the shell surface. The first two coats were stuccoed with sand of AFS fineness number 120 and the next four coats were with sand of AFS fineness number 42. After all coats, the shells were air dried for 24 hours. Two shells of each treatment were made. The pre-heated investment shells were poured with Ti-alloy.

2.2 Hot strength of ceramic shells

The dimensions of specimens are 25mm X 32mm X t mm, where t is the thickness of the shell. The specimens used for bending test are shown in figure 2. The test of hot modulus of rupture was conducted on the universal sand- strength testing machine with attached electrical oven as shown in figure 2. The temperature of the oven was measured with a thermocouple attached to it. To find hot modulus of rupture, the ceramic shell specimens were heated to various temperatures and the same was tested simultaneously in the oven for the bending strength.

2.3 % thermal expansion of ceramic shells

It was measured in terms of %volume expansion of the investment shells. The length, width and thickness of the shells were measured using vernier calipers before and after sintering in the electrical oven. The % thermal expansion was computed using the following formula:

$$\% \text{ thermal expansion} = \frac{V_2 - V_1}{V_1} \times 100$$

where, V_1 is the volume of the shell before sintering and V_2 is the volume of the shell after sintering.

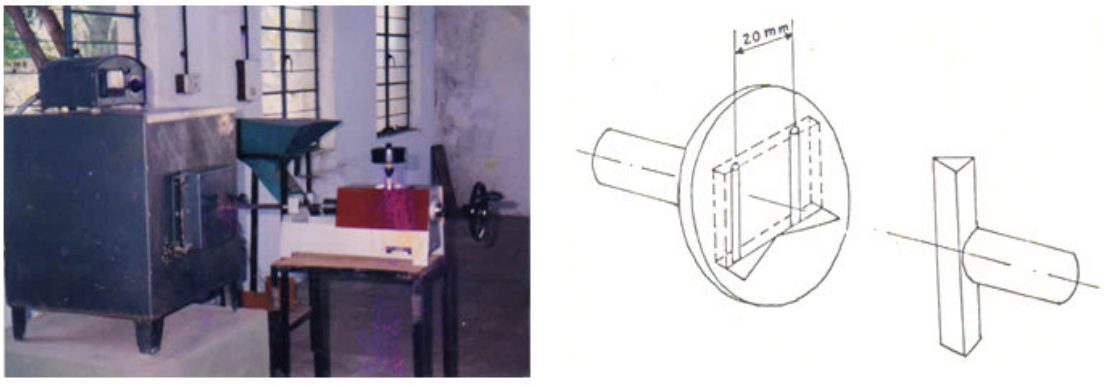

Figure 2. Hot bending strength test

2.4 Estimation of Metal-Mould reaction

Vickers hardness was carried out to find the hardness of surface layers of the castings. Scanning electron microcopy was carried out to characterize the fine-scale topography and establish the microscopic mechanisms governing metal-mould reaction. The scanning was carried in IICT (Indian Institute of Chemical Technology - Hyderabad) S-3000N Toshiba shows Scanning Electron Microscope.

3. Results and Discussion

The effect of sintering temperature on the hot bending strength of ceramic shells is shown in figure 3. The filler to binder ratio was 0.75 cc/ml. The hot bending strength zirconia shells decreases with increasing temperature [9]. This is due to phase transformation of zirconia at high temperatures. The phase diagram of carbon is shown in figure 1. Figure 4 shows the difference in the free energy of the low-temperature monoclinic and the high temperature tetragonal phases of zironia. The solid red line includes effects of thermal expansion. The results shown as dashed line use the computed lattice parameters at T=0 K. In the Raman spectra measured with increasing temperature (figure 5), the phonon frequencies of zirconia are seen as a function of temperature. The Raman active modes of monoclinic ZrO_2 which belongs to the space group C_{2h}^2 with four molecules per unit cell. After the temperature is increased from 1273 to 1473 K, the observed spectra change abruptly, this indicates a phase transition.

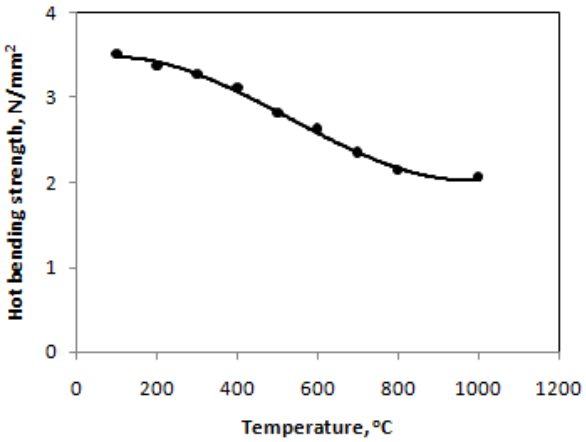

Figure 3. Effect of temperature on hot strength of ceramic shells.

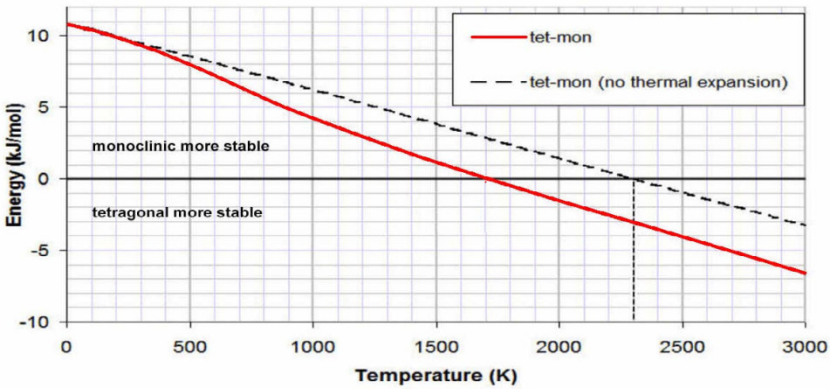

Figure 4. Difference in the free energy of the low-temperature monoclinic and the high temperature tetragonal phases of zironia.

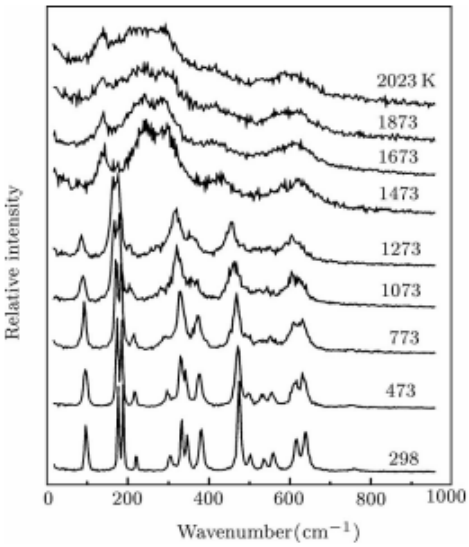

Figure 5. High temperature Raman spectra of monolithic and tetragonal phases of ZrO2

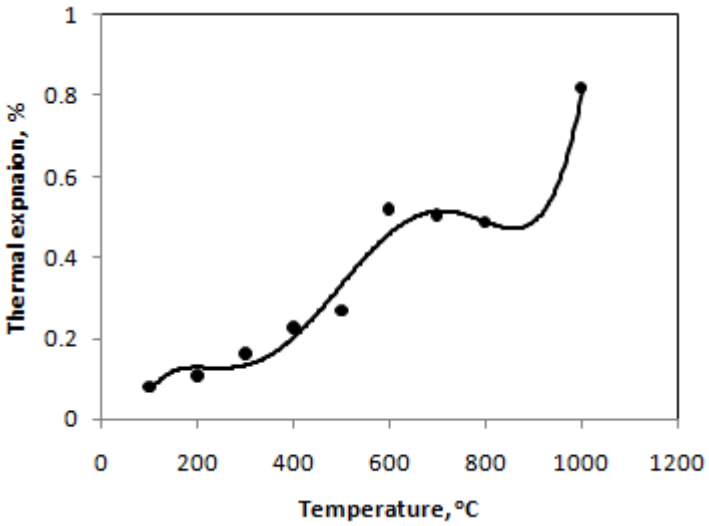

Figure 6. Effect of temperature on thermal expansion of shells.

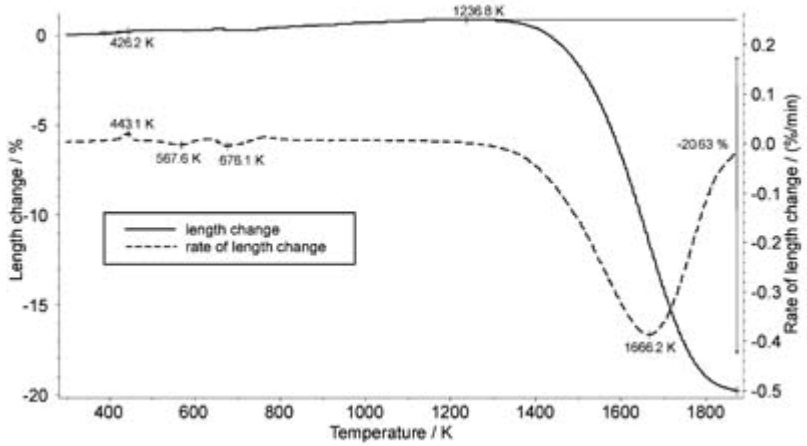

Figure 7. Effect of temperature on thermal expansion of zirconia.

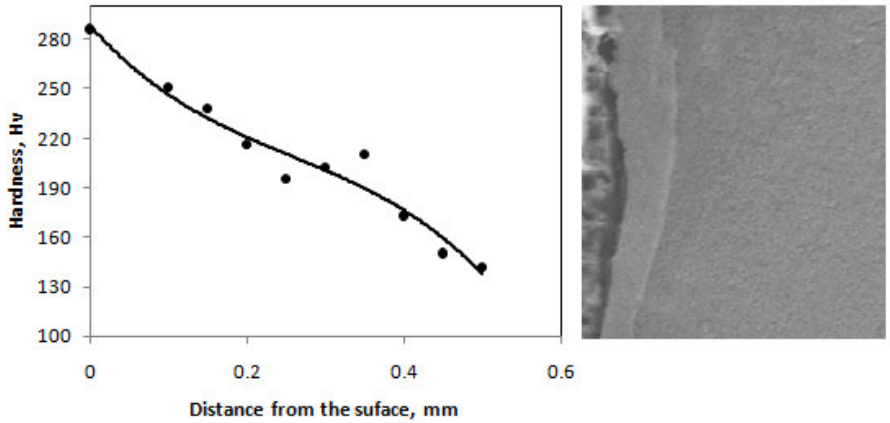

Figure 8. Metal-mould reaction (a) hardness variation and (b) formation of α phase

The thermal expansion curve for investment shells is illustrated in figure 6. The instability is on account of phase transition of zirconia, primary and back up sand used for sprinkling of shells [10]. This trend is confirmed with the thermal expansion of zirconia as shown in figure 7. Figure 8 shows the hardness profile of a sample from 25mm thick Zr-alloy 705

alloy casting made in zirconia investment shell mold. The hardness has a function of depth from the surface decrease with an increase in depth. This trend is indicative of presence of a hard ZrO_2 case layer (figure 8b) beneath the surface of the casting.

4. Conclusions

The hot bending strength fused silica shells increases with increasing temperature. The instability of thermal expansion curve is on account of phase transition of zirconia. A hard zirconia case layer is seen beneath the surface of the casting.

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