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Effect of sintering temperature on mechanical properties of magnesia partially stabilized zirconia refractory

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ABSTRACT

The optimized sintering conditions for a 3.5 wt% magnesia partially stabilized zirconia (Mg-PSZ) refractory were proposed in our recent research. The influence of the sintering temperature on the development of phase composition, microstructure, densification, thermal expansion and mechanical strength was studied in detail by X-ray diffraction (XRD), scanning electron microscope (SEM), Hepycnometer, high temperature dilatometry and three-point bending test. The samples sintered at 1670 °C had the highest bend strength, the maximum densification, the lowest thermal expansion coefficient (CTE), a homogeneous microstructure and a linear change in thermal expansion.

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1. Introduction

Vacuum induction melting (VIM) processing has been the primary melting method for nickel-based superalloy because it does not impact melt chemistry and gives results in good homogeneity of the melt [1]. But VIM is the only vacuum melting method which uses a refractory crucible made of oxides such as Al_2O_3 and MgO [2]. Unfortunately, the refractory is a contamination source of oxygen because of metal/refractory reactions leading to crucible disintegration. Oxygen is a harmful trace element that exists both in solid solution and oxide inclusions in the superalloy. Oxide inclusions can act as crack initiation sites and propagation paths, so it can dramatically affect the properties of the nickel-based superalloy [3]. Therefore, it is important to design an appropriate refractory to melt this superalloy. The stability of the refractory can be described by the following sequence:$Y_2O_3 > CaO > ZrO_2 > Al_2O_3 > MgO$ [4]. It is apparent that Y_2O_3 is the most stable crucible lining material. But Y_2O_3 is fairly expensive, which is a drawback [5]. CaO is not used because it is very susceptible to humidity [6] and therefore not suitable for crucible lining in industrial furnaces. MgO and Al_2O_3 crucible materials also cause a problem because of their unstability under high vacuum melting. The dissociation of these refractories leads to oxygen pick-up in the melt, which could eventually result in oxygen inclusion formation when the solubility limit is exceeded. Besides, the use of MgO crucible accelerates the formation of high melting inclusions ($MgAl_2O_4$) which deteriorate the cleanliness of the alloy [7]. Compared with refractories such as magnesia and alumina, zirconia ceramics show better chemical stability. Aneziris et al. [8] reported that the low porosity ZrO_2 based materials have been used in the near shape steel casting for high corrosion resistance. Hence, ZrO_2 is the most suitable refractory for melting nickel-based superalloy.

However, pure zirconia (ZrO_2) has three polymorphs depending on temperature: monoclinic (m) up to1170 °C, tetragonal (t) to 2370 °C, and above this temperature, cubic (c) [9,10]. After sintering at temperatures between 1500 and 1750 °C, pure zirconia ceramics break into pieces at room temperature [11]. Due to the destructive t→m phase transformation, pure zirconia is used quite rarely [12]. However, the effect of this phase transformation can be eliminated by doping zirconia with appropriate amount of oxides such as MgO and CeO_2 [13]. In view of the tremendous technological applications, PSZ sintering has received much attention from scientists and technologists. Mechanical properties can be enhanced by preparing PSZ ceramics that have high densities and small grain sizes after sintering [14]. Because of the different rates of pore closure, larger pores will develop into a porous microstructure during densification. These pores can be removed only at a high sintering temperature and a long sintering time [15]. Response surface methodology (RSM) was applied to determine the operational conditions for the properties of PSZ. The sintering temperature and heating rate have different effects on the densification and bending strength – sintering temperature is positive, heating rate is negative [16]. Although Young's modulus of the

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ZrO_2-3mol% Y_2O_3 does not change with sintering temperature, a slight decrease is observed in the hardness values above 1000 °C, which is attributed to microstructure coarsening [17].

The Mg-PSZ refractory has already become one of the most important materials because of its corrosion resistant, excellent chemical and thermomechanical properties [18]. Nevertheless, full exploitation of these advantages can only be realized if the final products have the required specifications comprising desired phase, density, microstructure and the bend strength [19]. Based on the equilibrium phase diagram MgO–ZrO_2 [20], the transformation temperature of t→m is about 1240 °C and the tetragonal and cubic solid solution becomes stable above 1400 °C. A feature of technological importance is the cubic and tetragonal solid solution phase field [8]. The crystal structure transformations involving volume changes exhibited thermal hysteresis, which may cause cracking defects. The hysteresis was controlled by several microstructure and chemical factors, such as grain size, tetragonal size, and the sintering process [21,22].

In this paper, the optimized sintering conditions for the 3.5 wt% Mg-PSZ refractory are proposed. The influence of the sintering temperature on the development of phase composition, microstructure, densification, thermal expansion and mechanical strength were studied in detail.

2. Experimental procedure

Commercial magnesia partially stabilized zirconia (3.5 wt% Mg-PSZ) powders (provided by Sunshine Capital Ltd, China) were selected as the starting material with a grain size distribution of 0–10 μm and d_{50}=2.7 μm. Solid discs (20 mm in diameter) and bars of 3.5 wt% Mg-PSZ were attained by slip casting. In order to form suspensions, the 3.5 wt% Mg-PSZ powders were dispersed in deionized water with an organic binder and dispersant, then milled for 4 h. The organic binder and dispersants were Arabic gum and Triethanolamine respectively. The stable suspensions with 78 wt% solids content were slip cast in plaster mold to obtain the green body. The green samples were sintered at different temperatures of 1600, 1620, 1650, 1670, 1690 °C with a dwell time at temperature of 4 h.

The crystal structure of samples sintered at different temperatures was characterized by XRD (D/MAX2200 V PC). Morphology was examined using a scanning electron microscope (SEM, Hitachi SU-1510). The amount of densification was obtained by the ratio of the densities of sintered samples determined using a He-pycnometer (AccuPyc II 1340) to the theoretical density. Differential scanning calorimetry analysis (DSC, Netzsch STA 449 F3) was employed to evaluate the phase transition temperature of 3.5 wt% Mg-PSZ powders in air from room temperature to 1350 °C. The bend strength of samples which were sintered at different temperatures was investigated using a three-point bending test with a span length of 30 mm on samples of 3 mm thickness × 4 mm width × 40 mm length. Five samples were tested to obtain the average data.

Linear shrinkage (dL/L_0) of the 3.5 wt%Mg-PSZ powders was measured in air using a push-rod dilatometer (Netzsch DIL 402C) from the room temperature to 1450 °C. The size of the sample bars for the dilatometer analysis was 8 mm length × 6 mm diameter.

3. Results and discussion

3.1. Sintering behavior of the 3.5 wt% mg-PSZ refractory

The sintering of green samples after drying took place in the dilatometer, which recorded the shrinkage value (dL/L_0) and the rate of linear shrinkage ($d(dL/L_0)$ of green bodies during heating.

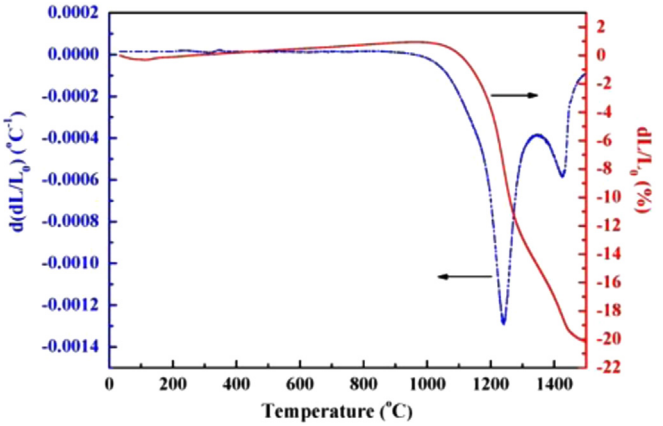

Fig. 1. Temperature dependence of shrinkage (dL/L_0) and shrinkage rate ($d(dL/L_0)/dT$) of the 3.5 wt% Mg-PSZ sample in the course of heating (5 °C/min).

The dynamic sintering curve of 3.5 wt% Mg-PSZ green body in air is presented in Fig. 1. It can be seen that there was no shrinkage below 1000 °C, but dramatically declined between 1000 °C and 1240 °C. The rapid shrinkage is caused by the formation of grain boundaries between the particles and a reduction in porosity. The maximum rate of shrinkage around 1240 °C was related to the phase transformation from monoclinic to tetragonal according to the equilibrium phase diagram MgO–ZrO_2 [20]. A large volume change is associated with this transformation, which was believed to be the reason for the catastrophic failure of zirconia ceramic [23]. It was noteworthy that the rate of shrinkage increased at 1426 °C, which may be caused by the phase transformation from tetragonal to cubic according to the phase diagram of MgO–ZrO_2. Thus, it was important to control sintering condition around this temperature. With increasing temperature, the rate of linear shrinkage gradually decreased.

The DSC curve of 3.5 wt% Mg-PSZ green samples is shown in Fig. 2. Two endothermic peaks occurred at 330 °C and 1240 °C respectively. Because decomposition temperature of Arabic gum and Triethanolamine are about 330 °C, the initial endothermic peak could be attributed to the decomposition of organics which were introduced during the slip casting. At this temperature, the effect of organics can be eliminated by prolonging soaking time during sintering. The other endothermic peak at 1240 °C corresponded to the phase transformation, which was consistent with the phase diagram and sintering curve.

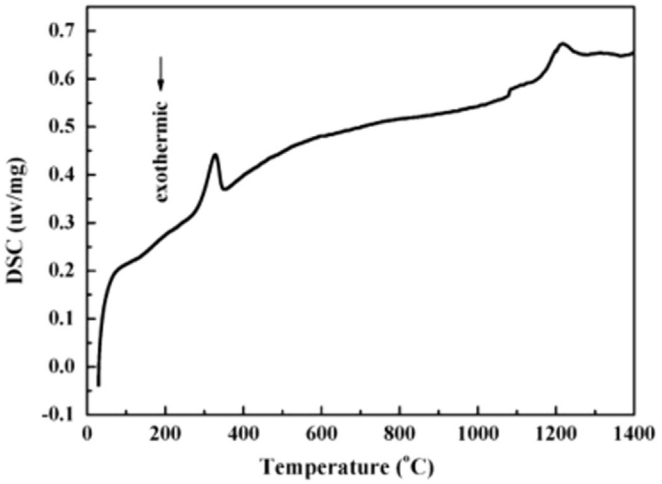

Fig. 2. Differential scanning calorimetry of 3.5 wt% Mg-PSZ green body.

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