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Effect of particle size distribution on properties of zirconia ceramic mould for TiAl investment casting

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Abstract: The effect of particle size distributions (PSDs) on the properties of zirconia ceramic mould for TiAl investment casting was described. The relationship between the zirconia powder characteristics and properties of the ceramic moulds was investigated. The particle size distribution, morphology of particles, viscosity of slurries, mechanical properties and fracture surfaces of ceramic moulds were examined. The effect of PSDs on the viscosity was observed through the measurement of slurries prepared from zirconia and binder. The morphology of the fracture surface of the zirconia moulds with different PSDs was also investigated. The measurement of bend strength shows that the mechanical behaviors of the green and fired zirconia ceramic moulds are comparable for all systems. The preliminary results illustrate that the PSDs play an important role in determining the quality of ceramic moulds and thus on the metallurgical quality of TiAl components produced by investment casting process.

Key words: particle size distribution; investment casting; TiAl; zirconia ceramic mould; properties

1 Introduction

Due to their low density, good corrosion resistance and interesting mechanical properties at high temperatures, γ -TiAl is widely regarded as a very demanding material for a wide range of potential applications in the aerospace system and automotive industry [1-2]. The production of TiAl components is expected to meet the new emerging markets through the newly developed processing techniques. Considering to the high brittle-ductile transition temperature and the tremendous oxygen affinity of TiAl, investment casting was selected over other routes for their potentially higher yields and greater cost effectiveness. However, the production of ceramic moulds is the most fundamental, demanding and complex steps in the investment casting process.

Until now, no refractory materials have been developed which are completely inert to the molten titanium [3–4]. Since the high aluminium content of TiAl alloys lowers their reactivity compared with titanium [5],

there has been a renewed interest in the investment casting refractory materials [6-7].

A lot of efforts were spent on the thermodynamic stability and interaction mechanism of refractory materials such as CaO [8–9], Al_2O_3 [10–11], ZrO₂ [12–13] and Y₂O₃ [11, 14–15] in contact with the molten TiAl alloys. Such information is of great interest not only for the purpose of solidification studies but also for the induction melting of TiAl alloys and their investment casting into the ceramic moulds as well. However, rarely systematic studies were conducted on the fundamental studies on the structure and properties of ceramic moulds which play an important role in the investment casting of TiAl alloys.

Previous studies indicated that zirconia is one of the least reactive materials and apparently holds promise as a mould refractory material usable in metallurgical processing and investment casting of TiAl alloys. In this work, zirconia sol is chosen as binder for the making of ceramic moulds with zirconia powders. The purpose of this paper is to outline a methodology of zirconia ceramic mould system suitable for the investment casting

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of TiAl alloys. The effect of particle size distribution on the mechanical properties and microstructure of the samples is investigated.

2 Experimental

Three CaO stabilized zirconia powders with different particle size distributions (PSDs) were used in this study: CSZ1, CSZ2 and CSZ3. The size distributions of these powders were measured with a Horiba LA-920 laser diffraction particle size analyzer.

The composition of slurry consists of zirconia sol, zirconia powder, wetting agent and anti-foam. The ceramic moulds within this study were designed to be representative of a standard moulds used for TiAl investment casting. Injected wax bars were used as formers for the ceramic moulds. The wax bars were dipped into the slurry. The wet patterns were then stuccoed with zirconia powders. This process was repeated several times until the total thickness of ceramic moulds reaches 5–7 mm.

A Hitachi S–570 scanning electron microscope was utilized to view the structure of zirconia particles and the cross-sections of ceramic moulds. Before the observation, the conductive sputter coating was performed on the surface of samples.

To evaluate the mechanical properties of the moulds at room temperature, three-point bend tests were conducted on the green and fired bars at room temperature. The samples were prepared upon a wax pattern. After dewaxing, the moulds were cut into rectangular test bars with dimensions of 40 mm \times 20 mm \times 4 mm according to the bending strength standard. And the test samples were fired at 950 °C for 2 h. The samples were loaded in an Instron 5569 test machine at a constant load rate of 0.5 mm/min until failure. All tests were conducted on five samples in order to verify the reproductivity of the test results.

The bending strength, σ_w , was calculated using Eq. (1):

$$\sigma_w = \frac{3FL}{2ah^2} \tag{1}$$

where F is the fracture load, L is the span length, a and h are the width and thickness of sample fracture area, respectively.

The load—deflection curve was converted into a stress—strain curve using Eqs. (1) and (2).

$$\varepsilon = \frac{6h\delta}{L^2} \tag{2}$$

where ε is the strain, and δ is the deflection.

3 Results and discussion

3.1 PSDs of particles

The particle size distributions of the powders are shown in Fig. 1. It can be seen from Fig. 1 that the median particle diameters are 20, 30 and 40 μ m, respectively. The sand CSZ3 has a broader particle size distribution while the sand CSZ1 presents a narrow particle size distribution among the three zirconia powders. The sand CSZ2 contains mixed powders size and narrower PSDs and would disperse better in the slurries. Because of TiAl's demands on surface quality of ceramic mould, it is necessary to use very tight quality control of raw materials and the slurries has to be applied to maintain the quality and for process stability. Further investigation of the particle size was carried out by SEM.



Fig. 1 Particle size distributions of zirconia powders

3.2 Morphologies of particles

The particle morphology of the zirconia powders observed by SEM is shown in Fig. 2. SEM observations confirm a substantial difference between the three samples. The particle size appears to be uniform (Fig. 2(a)). The particles show to be equiaxed. The particles of both large and small size mutually embed in each other. The appearance in Fig. 2(c) results from the wide PSDs. There exists big granules and smaller agglomerates. As shown in Fig. 2(b), the size distribution of zirconia is controlled so as to remove smaller ones, for the purpose of preventing agglomeration of zirconia was prepared by removing both fine and coarse particles. Therefore, it has medium distribution.

Some research results [16] indicate that the morphology of particle has a great impact on the quality of slurry in reproducing of tiny surface details and thin sections from ceramic moulds with small volumes of metal. It is clearly seen from the findings that the surface roughness of castings is directly related to the fineness of investment powders [17–18]. A decrease in the particle



Fig. 2 SEM micrographs of zirconia powder: (a) CSZ1; (b) CSZ2; (c) CSZ3

size of powders causes the formation of extremely small pores in the zirconia ceramic mould which prevents the penetration of molten TiAl into these cavities to a greater distance under the same hydrostatic or centrifugal pressure and thus improving the surface quality. However, decreasing particle size means increased specific surface area and affects the quality of castings reversely in terms of mechanical properties. It is known that, the finer the particle size, the smaller the pore diameter in the ceramic mould, which increases the difficulties in evacuating the air and the gases occupying the mould cavity throughout the mould wall, due to the low permeability during the melting process of TiAl alloys. These gases will finally remain in the TiAl castings in the form of gas porosity, leading to inferior mechanical properties.

3.3 Viscosity of slurries with different PSDs

The apparent viscosity as a function of shear rate for zirconia slurries with different PSDs is shown in Fig. 3.



Fig. 3 Apparent viscosity curves of zirconia slurries with different PSDs

These slurries show non-Newtonian flow behavior. In slips with both high and low PSDs (CSZ1 and CSZ3), the viscosity decreases significantly as the shear rate increases. Slips with intermediate PSD are relatively independent of shear rate. This observation is consistent with the behavior observed for blends of monodisperse spheres of different size (whose viscosity decreases on blending) [19]. Thus, we believe that our observation likely stems from blending colloids that have differences in particle size distribution, particle morphology, and surface forces.

3.4 Mechanical properties of ceramic moulds with different PSDs

The three-point bend test results of the green and fired moulds are shown in Fig. 4. CSZ2 exhibits higher green strength than that of the CSZ1 and CSZ3 mould, giving 4.27 and 2.67 MPa, respectively. A strength as high as 4.76 MPa for the green zirconia ceramic mould is attained, which exhibits the narrow PSD and median particle size. A similar fracture behavior was observed in fired systems (Fig. 4(b)). Both the green and fired stress—strain curves exhibit a strong PSDs dependence. These data offers some insights for adjusting the strength of zirconia ceramic moulds to resist cracking during dewaxing and handling process. The sufficient strength is big enough to survive during TiAl casting.

As seen from the data presented in Fig. 4, the bend strength of zirconia ceramic moulds is affected by two factors, namely, particle size and PSDs. The strength of CSZ2 having moderate PSD has the highest strength. A decrease in specific surface area decreases the contact surface of particles in moulding sands and thus decreases the strength. Although CSZ1 has the highest specific surface area (smaller particle size) than others,



Fig. 4 Stress—strain curves of zirconia mould prepared with different PSDs: (a) Green; (b) Fired

its bend strength is low than that of the CSZ2. This is most probably resulted from the higher porosity associated with lower stacking density.

3.5 Morphology of fracture surface

Figure 5 shows the microstructure of the broken surface of the green zirconia ceramic moulds with different PSDs. It can be easily visualized that larger particles exhibit irregular geometry, most of zirconia particles are nearly spherical in shape and some of them show close packing. Available data (Fig. 1) illustrated that the zirconia powder has a different PSDs. This indicates that the slurries containing median PSD can be accumulated to green compacts with a higher density. It is interesting to note that the green density of the zirconia mould acts as a function of PSD. All the slurries have certain viscosity, rather than having the lowest viscosity. This indicates that the rate at which the packing structure of the zirconia particles is built up is too fast to achieve a dense packing for the suspension having the lowest viscosity, although the zirconia particles slide much more easily over one another at the lowest viscosity than at higher viscosity. Therefore, it is more important for the slurries to have an optimal viscosity which is not too



Fig. 5 Morphologies of fracture surfaces of green zirconia ceramic moulds with different PSDs: (a) CSZ1; (b) CSZ2; (c) CSZ3

high for particle rearrangement and not too low for poor packing structure development.

An examination of the fractured surface for the fired body with different PSDs is shown in Fig. 6. The fired



Fig. 6 Morphologies of fracture surfaces of fired zirconia ceramic moulds with different PSDs: (a) CSZ1; (b) CSZ2; (c) CSZ3

ceramic derived from the median PSD shows a more uniform microstructure, particularly pore size and/or its distribution, than those of CSZ1 and CSZ3. Therefore, the lowest suspension viscosity associated with the stacking cast density may be considered as fully characterizing the final fired microstructure. Pore size as well as pore-size distribution within the ceramic moulds is critical factors governing the microstructure of ceramic moulds. The variation of the strength with respect to PSDs is believed to be due to the presence of residual porosity.

4 Conclusions

1) Slurries with median PSD and optimal viscosity facilitating particle arrangement and packing structure development is essentially important for obtaining a fine-grained, uniform sintered microstructure.

2) The bend strength of the zirconia ceramic moulds is directly related to the PSDs within the green and fired ceramic bars. It is recommended that the mechanical properties of zirconia ceramic moulds can be attributed to contact surface and residual porosity within the ceramic body. This work has shown that it is possible to produce stable zirconia slurries and ceramic moulds for investment casting of TiAl alloys.

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粉料的粒度分布对 TiAl 合金熔模精密铸造用 氧化锆陶瓷型壳性能的影响

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摘 要:研究粉料的粒度分布对 TiAl 合金熔模精密铸造用氧化锆陶瓷性能的影响。对粉料粒度分布、粉末形貌、 浆料黏度、型壳抗弯强度和断裂形貌之间的关系进行研究;通过测量氧化锆粉末和粘结剂构成的浆料的黏度来考 察粉末粒度分布对浆料黏的影响;对不同粒度分布氧化锆陶瓷型壳断口形貌进行观察。结果表明:粉料粒度分布 对未焙烧和焙烧后氧化锆陶瓷型壳的强度影响行为相似;粉料粒度分布对氧化锆陶瓷型壳的质量能产生很大的影 响并进而影响到熔模精密铸造制备 TiAl 合金构件的质量。

关键词: 粒度分布; 熔模精密铸造; TiAl 合金; 氧化锆陶瓷型壳; 性能

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