Rare Metal Materials and Engineering
Volume 45, Issue 2, February 2016
Online English edition of the Chinese language journal

Available online at www.sciencedirect.com
ScienceDirect

Cite this article as: Rare Metal Materials and Engineering, 2016, 45(2): 0303-0308.

ARTICLE

Mechanism and Influence of Re-dissolution of Zirconium Diacetate-Yttria Shell for Titanium Investment Casting

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Abstract: The re-dissolution of zirconium diacetate-yttria shell (ZA-Y shell) and its impact were investigated. The re-dissolution rate under moisture condition was measured by a balance. Three-point flexural strength was measured using a universal testing machine, and the fracture index was calculated. The distribution of the surface precipitates was observed by optical microscope, and its morphology and composition were tested using scanning electron microscopy (SEM) combined with energy spectrum analysis (EDS). The composition of the ZA-Y shell was measured by X-ray fluorescence (XRF). The results show that the re-dissolution is limited. The maximum water penetration rate is 2.5% and the dissolution rate is 0.8% within 20 min. The strength decreases by 26% and fracture index reduces to 66%. The surface precipitates are attributed to dissolution of ammonium metatungstate (AMT) and there are two reasons: drastic pressure release or fast drying condition with slow heat dissipation.

Key words: titanium; investment casting; re-dissolution; strength; precipitate

Because of the high pouring temperature and active chemical properties of titanium alloy, the facecoat shell in titanium investment casting must be made with special materials. A widely used facecoat slurry is usually made form zirconium diacetate binder, yttria powder and AMT (ammonium metatungstate) reinforcing agent[1]. The ZA-Y (zirconium diacetate-yttria) shell generally uses solvent dewaxing[2], but the toxicity of trichlorethylene is a threat to the workers and the environment, and a safe dewaxing method is necessary.

Autoclave dewaxing is a popular and environmentally friendly method. Compared to other methods such as hot water dewaxing and flash dewaxing, it has advantages for titanium complex structures[3]: little expansion stress to the shell[4], and the great heat transfer makes the wax flow out before big expansion; little increase to the internal stress[5], and the temperature is approximately 180 °C, lower than the decomposition temperature of zirconium diacetate and AMT.

However, the ZA-Y shell will dissolve in moisture environment. With the reduction of water, zirconium diacetate (Fig.1) polymerizes (Fig.2) and the binder gels gradually[6]. But in moisture environment, the coordination bond break, and the gelled zirconium diacetate dissolves again.

The re-dissolution may change the bonding condition of the shell, thus affecting its strength and surface quality. This would eventually lead to the inclusion defects caused by the peeling of facecoat or surface precipitates, which would significantly affect the quality of titanium castings. In the present paper, the re-dissolution of ZA-Y shell and its impact were studied. Water penetration and re-dissolution rate under water and steam condition were measured. Three-point flexural strength and fracture index under different conditions were compared. The morphology and the composition of the precipitates were investigated and the reasons were analyzed.

1 Experiment

Received date: January 07, 2015
Foundation item: National Natural Science Foundation of China (50875144)
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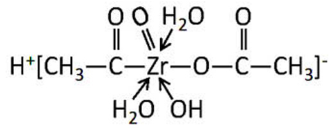

Fig.1 Chemical structure of zirconium diacetate

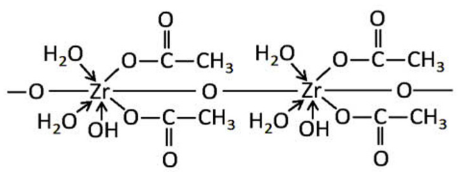

Fig.2 Chemical structure of polymerized zirconium diacetate

Zirconium diacetate binder, yttria powder and AMT were made into a slurry with the proper viscosity to make the ZA-Y layer, using yttria as sand material. Aluminum-silicon based slurry and sand were used to make the aluminum-silicon layer. The ZA-Y shell was made of 8.5 ZA-Y layers (8 layers and 1 seal), and the Y-Al shell was made of 2 ZA-Y layers and 5.5 aluminum-silicone layers. The drying time was 24 h/layer. To avoid the effects of wax infiltration, shells for re-dissolution and strength tests were separated from the wax by hand.

Re-dissolution and water penetration rate were measured with shell of 40 mm × 20 mm × 5 mm. The shell was dried at (105±5) °C to constant weight m_1, then immersed in (95±5) °C water (to avoid the boiling impact) for a certain time, which was weighed as m_2 and then dried to constant weight m_3. The re-dissolution rate S and water penetration rate W_m can be calculated by Eqs.(1) and (2):

$$S = \frac{m_1 - m_3}{m_1} \times 100\% \tag{1}$$

$$W_m = \frac{m_2 - m_3}{m_1} \times 100\% \tag{2}$$

Where, m_1 is mass before immersion, g; m_2 is mass after immersion, g; m_3 is mass after drying, g; S is re-dissolution rate, %; w_m is water penetration rate, %.

Strength properties were measured on a universal testing machine with the same shell, the fracture load and deformation were read by sensors, and the size was determined by caliper measurements. Three-point flexural strength M and fracture index FI[7] can be calculated by Eq.(3) and (4):

$$M = \frac{3Ls}{2wT^2} \tag{3}$$

$$\mathrm{FI} = \frac{M^2Tws}{18E} = \frac{L\gamma}{2} \tag{4}$$

Where, L is fracture load, N; s is span, 30 mm; W is shell width, mm; T is shell thickness, mm; E is modulus, MPa; γ is the deflection, mm.

The distribution of the surface precipitates was observed by optical microscope, then sampled by tape and tested using SEM combined with EDS after gilding to study the morphology and the composition. The composition of the ZA-Y shell was measured by XRF.

2 Results and Discussion

2.1 Re-dissolution of the ZA-Y shell

The re-dissolution under hot water and steam conditions was studied to learn the case in autoclave dewaxing. The re-dissolution degree of the two layers of direct contact with hot water would be more serious while the re-dissolution degree would be less but deeper under steam condition.

2.1.1 Re-dissolution in hot water

The re-dissolution and water penetration rate of the shell in hot water are shown in Fig.3. The penetration rate is comparable to that of the water-insoluble porous media, such as concrete and rock[8,9]. The penetration rate presents a nonlinear increase with time: it is high at beginning (10 min), but gradually slows down. The dissolution rate also owns similar nonlinear increase character. Within 20 min, the total water penetration rate is 2.5%, and the re-dissolution rate is 0.8%.

The re-dissolution of the shell is caused by the hydroxide ions which are absorbed onto the binder particle surface, resulting in the decomposition of polymeric zirconium diacetate. Not until the moisture penetrating to a certain depth, the re-dissolution of this depth occurs.

The penetration of porous media relates to solid content, porosity and pore size[9]. For the ZA-Y shell, the high content of yttria powder makes the surface density high. So its moisture penetration resistance is high, and the penetration rate is limited. Decomposition of zirconium diacetate will weaken the bonding between materials, but also form a high viscous fluid. This fluid will clog pores in the adjacent layer, further reducing the penetration rate. So the dissolution rate is confined, and the shell would not collapse immediately.

2.1.2 Re-dissolution in steam

The water penetration rate under atmospheric steam condition is shown in Fig.4. The penetration rate is 1.6%

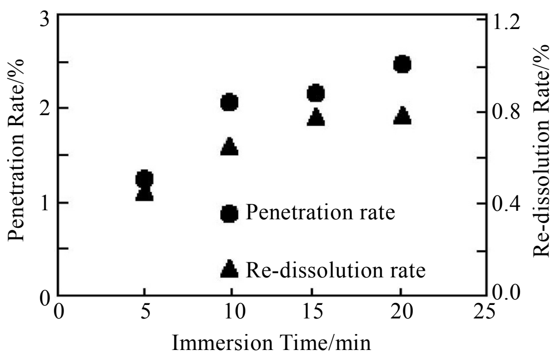

Fig.3 Re-dissolution and penetration rate vs immersion time