Effect of Trace O Element on High-temperature Wettability Between Ni₃Al Melt and Y₂O₃ Ceramic

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Abstract Effect of trace O element on the high-temperature wettability between Ni₃Al scrap melt and Y₂O₃ ceramic at 1873 K is investigated. With the increase in O content in Ni₃Al scrap melt from 6 to 21 ppm, the equilibrium contact angle decreases from 93.3° to 88.9°. The initial surface tension and adhesive work of molten Ni₃Al scrap drop from 3153.5 to 807.4 mN m⁻¹ and from 2974.6 to 882.2 mN m⁻¹, respectively. The average spreading rate increases from 0.03° s⁻¹ to 0.17° s⁻¹. The whole wetting processes are divided into four periods. The main driving force for spreading in period (1) is the adsorption and diffusion of active atoms around the interface. Trace O content in Ni₃Al scrap has a significant impact on interfacial reactions which mostly take place in the second stage, and can accelerate the spreading process on Y₂O₃ substrate. The reaction products during wetting process are Y₃Al₅O₁₂, YAlO₃ and Al₂O₃.

KEY WORDS: Ni₃Al scrap; Wetting; Interaction; Yttria; Oxygen content

1 Introduction

Ni₃Al-based superalloy is one of the widely applied single-crystal superalloys for turbine blades because of its high melting point ($T_m > 1573$ K) and superior high-temperature performance [1–3]. Rare noble elements such as Re and Hf are added to improve the high-temperature oxidation resistance and strength [4, 5]. During practical

production process, a large number of scraps, including sprues, casting riser and scrap castings, result in the low process yield (20–40%) of Ni₃Al superalloy. Most of these scraps have not been successfully reused, and plenty of the rare metal resources are wasted. Recently, in order to reduce the costs of the procedure and economize resources, recycling and reusing of the superalloy scraps are becoming increasingly important.

One of the main barriers to prevent superalloy scraps from being reused is the contamination of impurity elements in scraps. Some minority elements like O in the scraps are much higher than in normal parts. Rao et al. [6] have revealed that when O content in IN718 superalloy increased to 40 ppm, the rupture life and ductility under 690 MPa at 650 °C decreased by 27 and 25%, respectively. Moreover, trace O content can lead to inclusions and eventually create microcracks in castings [7].

Thus, the aim of purification melting technique of the scraps is to eliminate the influence of O contamination during vacuum induction melting (VIM). Oxide refractory

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crucibles, such as CaO and MgO crucibles, are applied during VIM. However, CaO is easily hydrated and MgO can be decomposed under high-vacuum and high-temperature environment [8, 9], leading O and other contaminations being introduced into the melt from crucibles. The purification effects of superalloy scraps are greatly diminished.

Recently, Y_2O_3 is studied and applied as refractory during VIM process because of its high thermodynamic stability and hydration resistance [10, 11]. Our previous work [12, 13] displayed that Y_2O_3 crucible showed a promising result during production of highly reactive Ti–Al- and Nb–Si-based superalloy, and the purification effects were significantly influenced by the wetting behaviors between superalloy melt and Y_2O_3 ceramic [14].

Besides that, O as surface active element has a great impact on the surface tension of the melt, influencing wettability between metal melt and ceramic. Matthias *et al.* [15] have investigated that the contact angle in Cu/ Al_2O_3 wetting system can vary between 125° and 22° when changing the oxygen partial pressure. Muolo *et al.* [16] have revealed that O can reduce the surface tension of liquid Ag when increasing the oxygen partial pressure. Thus, the O content in Ni_3Al scraps would also influence the purification effect. In order to promote the purification and reusing of Ni_3Al scraps, the mechanisms of the effect of O content on high-temperature wettability between Ni_3Al scraps and Y_2O_3 ceramic need to be studied. Unfortunately, so far, studies of the effect of O content on the high-temperature wettability between the superalloy scraps and the oxide ceramics are very limited and the corresponding mechanisms still have not been fully understood.

Therefore, in this work, we used modified sessile drop method to investigate the high-temperature wettability between Ni_3Al scraps with different O content and Y_2O_3 ceramic in high vacuum at 1873 K. The problems of pre-melting and pre-reaction were effectively avoided. The influences of different O contents on wettability of Ni_3Al scraps and Y_2O_3 substrate were studied. The interface was characterized, and interfacial reactions were investigated. The corresponding mechanisms of O content were clarified.

2 Experimental

Y_2O_3 ceramic substrate with ceramic powder in a diameter of 20 mm was made by direct compressing and sintering method. The surface roughness and open porosity of different substrates are strictly controlled to a close value. Ni_3Al scraps with the O contents of 6 ppm, 10 ppm and 21 ppm (wt%) were cut into 3 mm × 3 mm × 3 mm

cubes and grinded to remove the oxide layer. The contents of O in Ni_3Al scraps were measured by inert gas melting method (LECOONH836, USA). The nominal element compositions of this Ni_3Al superalloy used are shown in Table 1.

Wetting experiments were carried out in a modified sessile drop wettability testing apparatus (Fig. 1) under high vacuum at high temperature. The chamber was heated at the speed of 20 K min^{-1} to 1873 K. Y_2O_3 ceramic substrate was put on the sample carrier and adjusted to the horizontal position before evacuating and heating. In order to prevent the evaporation of elements in Ni_3Al superalloy under high vacuum, 1.1–1.2 atm argon (purity above 99.99%) was introduced into the chamber under the inner pressure of 5×10^{-4} Pa. Superalloy cube was dropped through an Al_2O_3 tube from alloy carrier to the surface of Y_2O_3 substrate, after the temperature in chamber stably reaching 1873 K. The pre-heating effects in conventional sessile drop method were successfully avoided, making the measurement of contact angle in this experiment more precise. Then, the whole wetting process was captured by a high-resolution CCD camera every 0.5 s. The time when the alloy cube totally melted was considered to be the starting time of the wetting process. After 20-min wetting at 1873 K, the chamber was slowly cooled down to room temperature. The pictures that were captured by the high-speed camera were analyzed by programs ADSA, SEDROPD and FTA32. Then, the data of contact angle and surface tension were obtained.

After the wetting experiment, the droplet and substrate were cut and observed by scanning electron microscopy (SEM, CamScan-3400, UK). Surface roughness and surface 3D visualization were obtained by Confocal Scanning Laser Microscopy (CSLM, LEXT OLS4100, Japan). Phases of reaction products at the wettability interface were identified by X-ray microdiffraction (Micro-Area XRD, D8 DISCOVER With GADDS, Germany). Element compositions were analyzed under an energy-dispersive spectrometer (EDS, Oxford INCAPentaFET-X3, UK).

3 Results

3.1 Wetting Kinetics

The variation of contact angle and drop base radius with time for the molten Ni_3Al superalloy with different O

Table 1 Nominal element compositions of Ni_3Al superalloy (wt%)

Al	Mo	Ta	Cr	Ni
7.6–8.3	9.0–13.0	2.4–4.0	1.5–2.5	Bal.

Fig. 1 Schematic of modified sessile drop wettability testing apparatus

contents on the Y_2O_3 surfaces at 1873 K is shown in Fig. 2. The time for reaching equilibrium becomes shorter with the increase in O contents. Equilibrium time is 777, 550 and 270 s for Ni_3Al scrap melt with O contents of 6, 10 and 21 ppm, correspondingly. The rising of O content in the melt can accelerate the spreading during wetting process. Therefore, it will reduce the mold filling time when increasing the content of O in the melt during directional solidification process or casting process. The average spreading rates in the whole wetting processes are 0.03, 0.07 and $0.17^\circ s^{-1}$, respectively. These wetting processes can be roughly divided into four periods according to the shape of wetting curve: (1) rapid spreading period ($0 < t < t_1$); (2) quasi-linear spreading period ($t_1 < t < t_2$); (3) steady spreading period ($t_2 < t < t_3$); (4) equilibrium period ($t > t_3$) (t : holding time; t_1 , t_2 , t_3 : different times in the wetting curve). In period (1), the molten droplet spreads rapidly and the contact angle drops very sharply. However, in period (2), the driving force of spreading process declines and the spreading rate decreases. The variation of contact angle versus time in this stage is called ‘quasi-linear’ stage by other researchers [17]. In period (3), the wetting and spreading processes continue, but at a very low rate. This stage, in which the change of the contact angle is very limited (less than 5°), takes place at a spreading rate lower than that in other periods. Finally, in period (4), the whole system steadily reaches thermodynamic and kinetic equilibrium.

Plots of variation of initial contact angle and equilibrium contact angle with O contents in Ni_3Al/Y_2O_3 wetting system are shown in Fig. 2d. The wetting process shows a different behavior in the alloy with different O contents. The initial contact angle increases a lot, from 119.6° to 135.6° with the rise in O contents from 6 to 21 ppm. Meanwhile, the equilibrium contact angle decreases from 93.3° to 88.9° . When the equilibrium contact angle decreases, the wettability between Ni_3Al scraps and Y_2O_3 ceramic has been improved. Accordingly, the capillary holes on the surface of Y_2O_3 ceramic are more likely to be fulfilled by superalloy melt. Moreover, localized thermal stress will be produced around the fulfilled capillary holes. Consequently, some Y_2O_3 ceramic particles will fall off and cause the contamination of the melt.

3.2 Initial Surface Tension and Initial Adhesive Work

The plot of the variation of initial surface tension with different O contents in Ni_3Al superalloy scraps is given in Fig. 3a. According to Dupre equation:

$$w_{ad} = \gamma_{lv} \cdot (1 + \cos \theta), \quad (1)$$

where w_{ad} is the adhesive work, γ_{lv} is the surface tension of liquid Ni_3Al , and θ is the dynamic contact angle. Respective initial adhesive work is calculated and given in Fig. 3b. The initial surface tension of molten Ni_3Al scraps drops from 3153.5 to $807.4 mN m^{-1}$. Meanwhile, the

Fig. 2 Variation of contact angle and drop base radius with time for molten Ni_3Al superalloy with O contents of 6 ppm **a**, 10 ppm **b**, 21 ppm **c** variation of initial contact angle and equilibrium contact angle with different O contents in Ni_3Al superalloy scraps at 1873 K **d**

Fig. 3 **a** Initial surface tension and **b** initial adhesive work in $\text{Ni}_3\text{Al}/\text{Y}_2\text{O}_3$ wetting system with different O contents at 1873 K

initial adhesive work dramatically goes down from 2974.6 to 822.2 mN m^{-1} . Both of the initial surface tension and initial adhesive work are heavily influenced by the O contents in Ni_3Al scraps melt.

3.3 Interaction Surface Characterization

According to the results of XRD results shown in Fig. 4d, the reaction products around $\text{Ni}_3\text{Al}/\text{Y}_2\text{O}_3$ interface (Fig. 4a–c) include $\text{Y}_3\text{Al}_5\text{O}_{12}$, YAlO_3 and $\alpha\text{-Al}_2\text{O}_3$. With

different O contents, the reaction products between Ni_3Al superalloy scraps and Y_2O_3 substrate remain the same. Although new reaction products are formed at $\text{Ni}_3\text{Al}/\text{Y}_2\text{O}_3$ interface, the matrix of superalloy remains clean. Figure 5 shows the microstructures of Ni_3Al scraps after wetting and solidification.

As for the surface microstructure of Y_2O_3 substrate (Fig. 6), it is slightly influenced by the wetting process at 1873 K. Before wetting, the densities of Y_2O_3 substrates are high except for some open pores. The average open porosity of Y_2O_3 substrates is 0.73%. Most Y_2O_3 particles were sintered together and were in the same horizontal plane after pressing and sintering. After wetting process, the flatness of Y_2O_3 substrates remains nearly still. However, there are new reaction products including $\text{Y}_3\text{Al}_5\text{O}_{12}$, YAlO_3 and Al_2O_3 on the surface of the ceramic, causing the slight change in surface microstructure. The falling degree of surface ceramic particles caused by molten Ni_3Al superalloy on Y_2O_3 substrate is relatively low since no

evident dents and pits are found in the area covered by Ni_3Al scrap melt.

4 Discussion

Under ideal conditions (pure liquid, smooth and inert substrate), the driving force in the wetting process can be expressed as the following equation:

$$F_d(t) = \sigma_{sv} - \sigma_{sl} - \sigma_{lv} \cos \theta(t), \quad (2)$$

where $F_d(t)$ is the driving force, σ_{ij} ($i, j = s, l, v$) is the characterized interface energy in the system, $F_d(t) = 0$ when reaching balance, which is Young's equation.

However, in the reactive wetting system, the driving force is influenced by chemical reaction, adsorption, dissolution and diffusion [18]. According to the adsorption-controlled theory [19, 20] and interface reaction free

Fig. 4 Wetting interfaces between Y_2O_3 substrates and Ni_3Al superalloy scraps with O contents of 6 ppm **a**, 10 ppm **b**, 21 ppm **c** wetting interface phase compositions of different O contents containing system **d**

Fig. 5 Microstructures of Ni_3Al scraps with O contents of 6 ppm **a**, 10 ppm **b** and 21 ppm **c** after wetting on Y_2O_3 substrate at 1873 K

Fig. 6 SEM **a, b** and CSLM 3D visualization **c, d** images of surface microstructures of Y_2O_3 substrate before **a, c** and after **b, d** wetting

energy change-controlled theory [18], all the factors which influence the driving force can influence the contact angle.

To discuss the kinetics during wetting process in systems with different O contents, the first three periods before reaching equilibrium are fitted and compared. The average spreading rates in period (1) could be represented by the slope of the linear fitting curve (Fig. 7a), which are 0.17, 0.44 and $1.39^\circ \text{ s}^{-1}$ in systems with O contents of 6, 10 and 21 ppm, correspondingly. The spreading rate increases with the rise in O content in this period. Meanwhile, the duration of period (1) becomes shorter. Moreover, the initial surface tension and initial adhesive work of Ni_3Al scrap melt decline with the rise in O content. Adhesive work is used to describe bonding strength at the interface

between the liquid and solid phase [21]. When the wetting process begins, the surface free energy (represents 1 mol liquid spreading on an atomic thickness) would vary with time. According to interface reaction free energy change-controlled theory [22], there is an equation to express the relation between adhesive work (w_{ad}) and surface free energy change ($w_{\text{a} \times \text{mol}}$):

$$w_{\text{a} \times \text{mol}} = (M/\rho)^{2/3} \times N^{1/3} \times f \times w_{\text{ad}} / (4.19 \times 10^7), \quad (3)$$

where M is the molecular weight of the Y_2O_3 ($225.82 \text{ g mol}^{-1}$), ρ is the density of the Y_2O_3 substrate (4.48 g cm^{-3}), N is the Avogadro's constant (6.02×10^{23}), and f is the packing coefficient (≈ 1 [23]).

Fig. 7 **a** Variation of contact angle with time in period (1) and **b** variation of $\ln(\cos\theta_F - \cos\theta)$ with time in period (2) (θ_F : equilibrium contact angle)

The calculated results of w_{ad} and $w_{a \times mol}$ in period (1) are listed in Table 2.

As revealed by Naidich et al. [22], the intermolecular bond energy is below the order of 10 kcal mol^{-1} , and that of chemical bond energy is above the order of $100 \text{ kcal mol}^{-1}$. For the wetting process from the beginning of total melting of Ni_3Al to the end of period (1), the surface free energy change remains in the range from 22.58 to $86.24 \text{ kcal mol}^{-1}$. It means that chemical interaction has already occurred since the beginning of the wetting process. However, the intermolecular bond energy is in the dominant position. It shows that the adsorption of the active atoms at the interface is the main driving force for wetting and spreading in this stage. What's more, the active atoms here are mainly Al rather than O in the Ni_3Al scrap melt. It is generally believed that O has a significant effect on reducing surface tension in liquid metals [15, 16]. Therefore, the initial surface tension, initial adhesive work and even initial surface free energy change ($w_{a \times mol(0)}$) of liquid Ni_3Al superalloy have been negatively correlated with the increase in O content in the results. The effects of O content in period (1) are mainly related to these

parameters. But $w_{a \times mol(t)}$ in the first period is not significantly correlated with O content. In $\text{Ni}_3\text{Al}/\text{Y}_2\text{O}_3$ wetting system, we presume that the transmission of Al atoms to the interface is the main reason for causing the difference of $w_{a \times mol(t)}$, because Al is also one major surface active element [24] in Ni_3Al system.

As for period (2), according to the representative equation of reaction-controlled spreading model proposed by Dezellus et al. [17]:

$$\cos\theta_F - \cos\theta = (\cos\theta_F - \cos\theta_0) \exp(-kt), \quad (4)$$

where θ_0 and θ_F are initial contact angle and equilibrium contact angle, and k is the kinetic constant which represents the spreading rate and could be calculated from the slope of the linear fitting curve of $\ln(\cos\theta_F - \cos\theta)$ versus time. As shown in Fig. 7b, k is 7.19×10^{-3} , 9.65×10^{-3} and $1.17 \times 10^{-2} \text{ s}^{-1}$ in 6 ppm, 10 ppm and 21 ppm O content systems. Just like in period (1), period (2) also shows a tendency of fast spreading with the increase in O content. The good agreement between the variation of contact angle and reaction-controlled spreading model reveals that, in the second period, the wetting and spreading processes are mainly controlled by the interfacial reaction.

Table 2 Adhesive work and surface free energy change in period (1)

O content	Holding time (s) t	Adhesive work (mN m^{-1})		Surface free energy change (kcal mol^{-1})	
		$w_{ad(0)}$	$w_{ad(t)}$	$w_{a \times mol(0)}$	$w_{a \times mol(t)}$
6 ppm	88.1	2974.6	2629.7	81.67	72.21
10 ppm	65.7	1325.9	3140.8	36.41	86.24
21 ppm	15.0	822.2	2346.7	22.58	64.43

* t is holding time of wetting process, $w_{ad(0)}$ and $w_{ad(t)}$ are adhesive work of the melt when $t = 0$ or other value, $w_{a \times mol(0)}$ and $w_{a \times mol(t)}$ are the surface free energy changes of the interface when $t = 0$ or other value

Y_2O_3 has a strong thermodynamic stability that hardly dissolute into Ni_3Al melt under 1873 K [25]. However, new phases of $\text{Y}_3\text{Al}_5\text{O}_{12}$, YAlO_3 and Al_2O_3 were formed. Reactions as followed can take place [26]:

$\text{Y}_3\text{Al}_5\text{O}_{12}$ and YAlO_3 are the compound oxides. They are both bonded by different ratios of Y_2O_3 and Al_2O_3 [27, 28]. These reactions are limited by the transmission of $[\text{Al}]$ and $[\text{O}]$. Since Y_2O_3 does not dissolute, the diffusion process is unidirectional. The schematic of $[\text{Al}]$ and $[\text{O}]$ diffusion from molten Ni_3Al superalloy to the solid–liquid two-phase zone is shown in Fig. 8. According to Fick's law, the diffusion rate depends on the concentration of $[\text{Al}]$ and $[\text{O}]$. The content of Al is rich enough in Ni_3Al superalloy. The contents of O are 6, 10 and 21 ppm in the melt, respectively, and the corresponding molar concentrations are 2.99, 4.99 and 10.49 mol ml^{-1} . (The density of Ni_3Al superalloy is 7.99 g cm^{-3} .) So reactions 5–7 are strictly limited by the transmission of $[\text{O}]$ from the melt to the solid–liquid interface. At the beginning of the wetting process, the variation of surface tension as a function of oxygen concentration in the liquid phase can be expressed by the Gibbs adsorption equation [29]:

$$\Gamma_{\text{O}}^{\text{sl}} = -C_{\text{O}} \cdot d\gamma_{\text{sl}} / (RTdC_{\text{O}}), \quad (8)$$

where $\Gamma_{\text{O}}^{\text{sl}}$ is the amount of oxygen adsorption at the solid–liquid interface, C_{O} and γ_{sl} are the oxygen concentration and surface tension of solid–liquid interface, R and T are the ideal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$) and temperature, and $d\gamma_{\text{sl}}/dC_{\text{O}}$ could be calculated by the slope of the linear fitting curve of γ_{sl} versus C_{O} as shown in Fig. 9. Accordingly, $\Gamma_{\text{O}}^{\text{sl}}$ is 5.13×10^{-5} , 8.57×10^{-5} and $1.80 \times 10^{-4} \text{ mol m}^{-2}$ in 6, 10 and 21 ppm O content system. These values presented here are in good agreement with those found in other investigations, which are in the order of $10^{-5} \text{ mol m}^{-2}$ [29, 30].

Fig. 9 Variation of initial surface tension with C_{O} at 1873 K in Ni_3Al superalloy scraps

With the increase in O content, the adsorption of O from superalloy melt to solid–liquid interface has increased more than three times for systems with O contents from 6 ppm to 21 ppm. When period (2) begins during the wetting process, more $[\text{O}]$ are transferred to the solid–liquid two-phase zone in system with O content of 21 ppm than others. Reactions 5–7 could take place more easily because the concentration of reactants has influence on the chemical equilibrium. As the chemical reaction is one major factor which promotes wetting process [31], the kinetic constant k in period (2) becomes larger with the increase in O content in the melt. Meanwhile, the duration time of period (2) is shortened. The effect of O content on the wetting process in period (2) is mainly through the change of the chemical reaction rate around the solid–liquid interface.

Like in period (1), the average spreading rate in period (3) could also be represented by the slope of the linear fitting curve. The average spreading rate is 0.003, 0.005 and 0.014 s^{-1} in systems with O contents of 6 ppm, 10 ppm and 21 ppm. The spreading rates decrease a lot compared with that in period (1). In this period, both the diffusion of mass and chemical reaction around interface

Fig. 8 Mechanism and direction of diffusion and adsorption around $\text{Ni}_3\text{Al}/\text{Y}_2\text{O}_3$ wetting interface in different O content systems

gradually tend to an end. When the whole system reaches the kinetic and thermodynamic equilibrium in period (4), the wetting process stops.

5 Conclusions

The high-temperature wettability between Ni₃Al scrap melt and Y₂O₃ ceramic at 1873 K is investigated. Based on the results, effect of trace O content on the wetting and spreading behaviors was discussed. The main conclusions are as follows:

1. With the increase in O contents in Ni₃Al superalloy scraps from 6 ppm to 21 ppm, the equilibrium contact angle decreases from 93.3° to 88.9°. The initial surface tension of Ni₃Al scrap melt drops from 3153.5 to 807.4 mN m⁻¹. Initial adhesive work decreases from 2974.6 to 882.2 mN m⁻¹.
2. The average spreading rate of molten Ni₃Al scraps on Y₂O₃ substrate increases from 0.03 to 0.17° s⁻¹. The rise in O content in Ni₃Al scraps melt can accelerate the spreading rate on Y₂O₃ substrate.
3. The main driving force for spreading in period (1) is the adsorption and diffusion of active atoms around the interface. The effect of O content on promoting spreading is mainly performed by controlling chemical reactions which mostly take place in period (2). The reaction products are Y₃Al₅O₁₂, YAlO₃ and Al₂O₃.

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