[Article ID] 1003- 6326(2002)04- 0656- 03

Effect of high temperature aging on microstructure and mechanical properties of a directionally solidified Ni_3Al base alloy IC6A①

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[Abstract] The effect of aging at 950 ℃ for up to 2 000 h on microstructure and mechanical properties of alloy IC6A was investigated with scanning electron microscopy(SEM), transmission electron microscopy(TEM) and energy dispersive spectroscopy(EDS) of electron probe micro-analyzer(EPMA). The results show that Ni_3Y and $Mo_{1.24}Ni_{0.76}$ precipitated in the interdendritic area due to the addition of yttrium have no obvious change during high temperature aging. A needle like or rod like phase named *Y*-NiMo precipitated after aging for about 200 h. The amount and size of the *Y*-NiMo phases increase with the increase of aging time, which is similar with that in alloy IC6. The ultimate tensile strength at room temperature and stress rupture life under 1 100 ℃, 90 MPa of alloy IC6A have no obvious change before aging for 1 000 h and decrease obviously with increasing aging time after aging for 1000h. The yield strength of alloy IC6A at room temperature decreases obviously after aging for 500h and however, has no obvious change during further aging.

[Key words] nickel aluminide; mechanical property; microstructure
[CLC number] TG 132.3 **[Document code]** A

1 INTRODUCTION

The directionally solidified Ni_3Al base alloy IC6A with the chemical composition of Ni-(7.5~8.5)Al-(13.0~15.0)Mo-(0.02~0.1)B-(0.005~0.05)Y(mass fraction, %) has been recently developed based on alloy IC6 as a high-temperature structural material used for advanced jet-engine vanes operating in the temperature range of 1 050 ~ 1 100 ℃[1, 2]. Alloy IC6A not only has high yield strength and fairly good ductility from room temperature to 1 100 ℃, but also has high creep resistance in the temperature range of 760~ 1 100 ℃. Similar to alloy IC6, alloy IC6A has low density(7.9 g/cm^3), low cost, high incipient melting temperature (1 300 ℃). Compared with alloy IC6, the high temperature oxidation resistance of alloy IC6A is substantially improved due to the presence of proper amounts of yttrium[3~6], which is very important for the industrial application of the alloy. In the present investigation, the effect of aging at 950 ℃ for different time up to 2 000 h on microstructure and mechanical properties of alloy IC6A was studied

2 EXPERIMENTAL

The master alloy ingot with a diameter of 76~80 mm was prepared in a vacuum induction furnace at 1 580~ 1 600 ℃ for 20~ 30 min, then remelted in MgO crucible in a commercial vacuum induction furnace and directionally solidified (DS) to produce columnar grain specimens with the size of 14~ 16 mm in diameter and 150~ 180 mm in length. The drawing rate of the Al_2O_3 precision investment mould filled with molten alloys, and connected to a water cooled copper plate, was 6~ 8 mm/min. The temperature of the mould was kept in the range of 1 490~ 1 520 ℃. The as-cast specimens were homogenized at 1 260 ℃ in air for 10 h followed with oil-quenching.

The size of the specimens for stress rupture and tensile tests is 5 mm in diameter and 25 mm in gauge length. The stress rupture tests were carried out at 1100 ℃ in air by using constant load creep machines, with the testing temperatures controlled within ±5 ℃. The tensile properties were examined at room temperature in air in an INSTRON-4507 machine with constant displacement method. The strain rate of specimen before and after yielding are 1 mm/min and 5 mm/min, respectively.

The specimens for microstructural analysis were polished and etched in the solution of 25% nitric acid + 50% hydrofluoric acid + 25% glycerin (volume fraction) and then examined in a JSM-5600 LV scanning electron microscope (SEM) and in a JXA 8600 electron probe micro-analyzer(EPMA). The thin foils for transmission electron microscope (TEM) study were done by twin-jet electrolytic polishing using 17% perchloric acid + 83% alcohol solution (volume fraction) under conditions of about 90 V at − 20 ℃ or below and then examined in a JEM 2010 transmission electron microscope under 200 kV.

3 RESULTS AND DISCUSSION

3.1 Effect of aging on microstructure

The microstructure of alloy IC6A examined by

① [Foundation item] Project (715- 005- 0020) supported by the National Advanced Materials Committee of China
[Received date] 2001- 10- 08

SEM after homogenization at 1 260 ℃ for 10 h followed by oil quenching can be divided into interdendritic and dendritic areas, as illustrated in Fig. 1. The volume fraction of γ phase is about 20% ~ 25% and that of γ′ phase is about 80% ~ 75%. The dimension of γ′ phase is 0.1~ 0.3 μm in interdendritic area and 1~ 3 μm in dendritic area. Borides usually appear in the interdendritic area when boron content exceeds 0.16% (mole fraction). When the content of yttrium in alloy IC6A is controlled in the upper limit (⩾ 0.03%, mass fraction), bulk shape regions appear in the interdendritic area surrounded by large size γ′ precipitates, as shown in Fig. 2. The conventional TEM and EDS analysis results show that the bulk shape regions consist of γ phase, Ni_3Y and $Mo_{1.24}Ni_{0.76}$ as well[7]. Fig. 3 shows the dark field TEM images of Ni_3Y and $Mo_{1.24}Ni_{0.76}$.

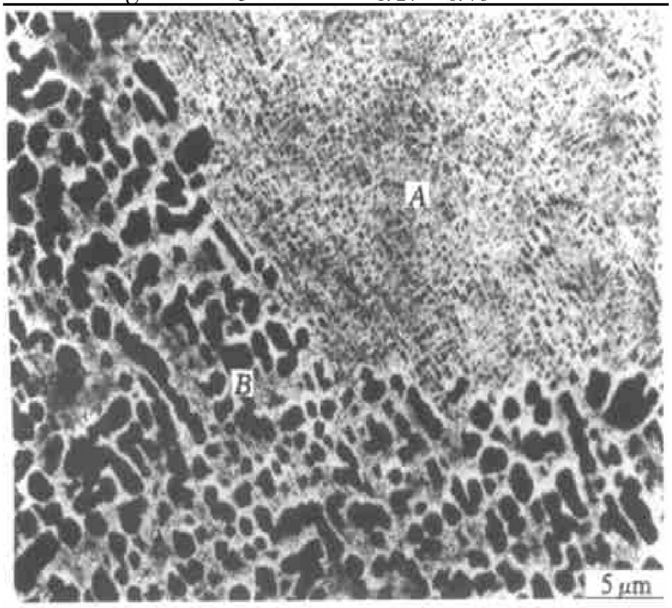

Fig. 1 SEM image of homogenized IC6A alloy
A —Interdendritic area; *B* —Dendritic area

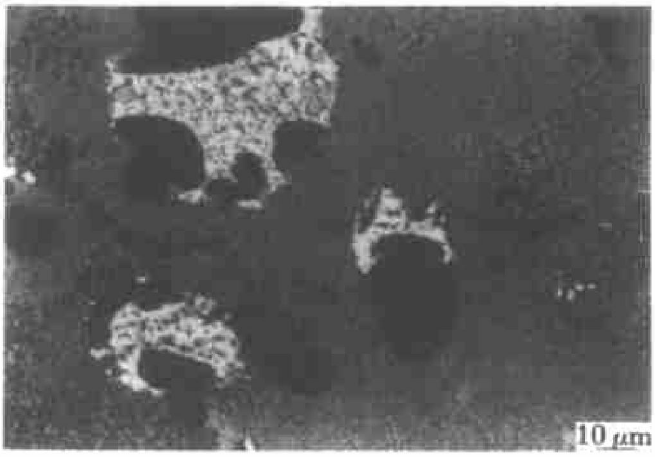

Fig. 2 Back scattered electron image (BSEI) of alloy IC6A with upper limit of yttrium showing bulk shape regions

There is no obvious change in microstructure of alloy IC6A after aging at 950 ℃ for less than 200 h, especially in the region consisted of γ phase, Ni_3Y and $Mo_{1.24}Ni_{0.76}$, as illustrated in Fig. 4. After aging

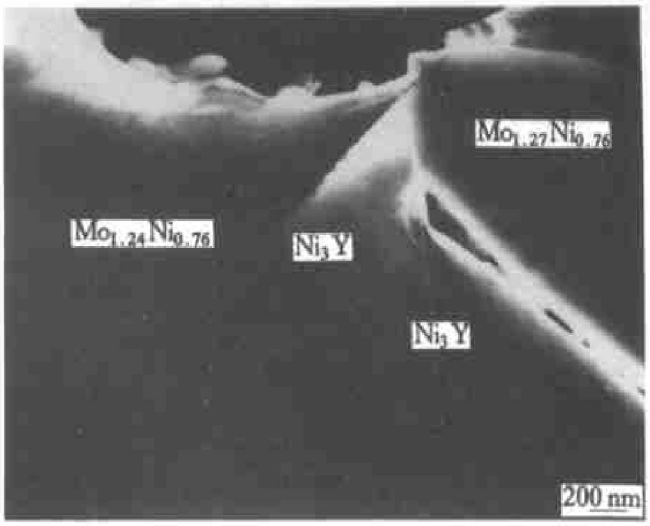

Fig. 3 Dark field TEM image of Ni_3Y and $Mo_{1.24}Ni_{0.76}$

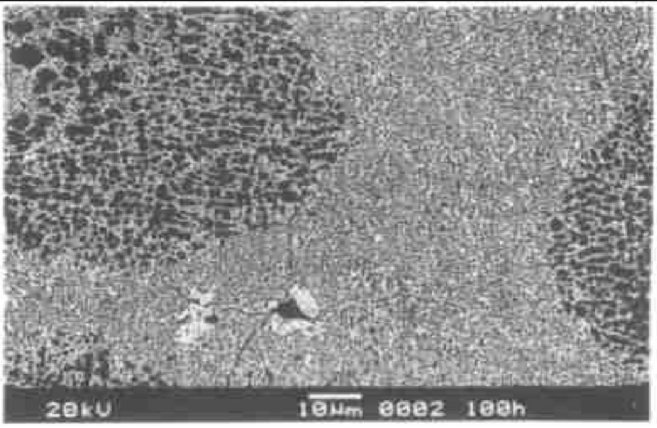

Fig. 4 Typical back scattered electron image (BSEI) of alloy IC6A after aging at 950 ℃ for less than 200 h

for over 200 h, the main change in microstructure of alloy IC6A is the precipitation of a needle or rod like phase, as shown in Fig. 5, which gives the typical microstructure of alloy IC6A after aging for 400 h and 2 000 h respectively. The conventional TEM and EDS analysis results show that the needle or rod like phase has the same crystal structure and chemical composition as *Y*-NiMo phase precipitated in alloy IC6 after aging at 900~ 1 150 ℃ for different time[8]. The size and volume fraction of *Y*-NiMo phase in alloy IC6A increase with increasing aging time after aging for 200 h. In alloy IC6, *Y*-NiMo phase precipitates during the early stages of aging (for example 25 h at 900 ℃, 1 000 ℃ and 1 100 ℃)[8], while in alloy IC6A, *Y*-NiMo phase precipitates after aging for over 200 h at 950 ℃, which is caused by two reasons. First of all, the presence of yttrium can inhibit the inter-diffusion speed of elements like Ni and Mo between interdendritic and dendritic regions, and hence retard the precipitation of *Y*-NiMo phase. Secondly, the formation of $Mo_{1.24}Ni_{0.76}$ in the interdendritic region consumes Ni and Mo, which decreases the saturation of Ni and Mo in the regions that *Y*-NiMo phases are formed during aging. From Fig. 5 it also can be seen that

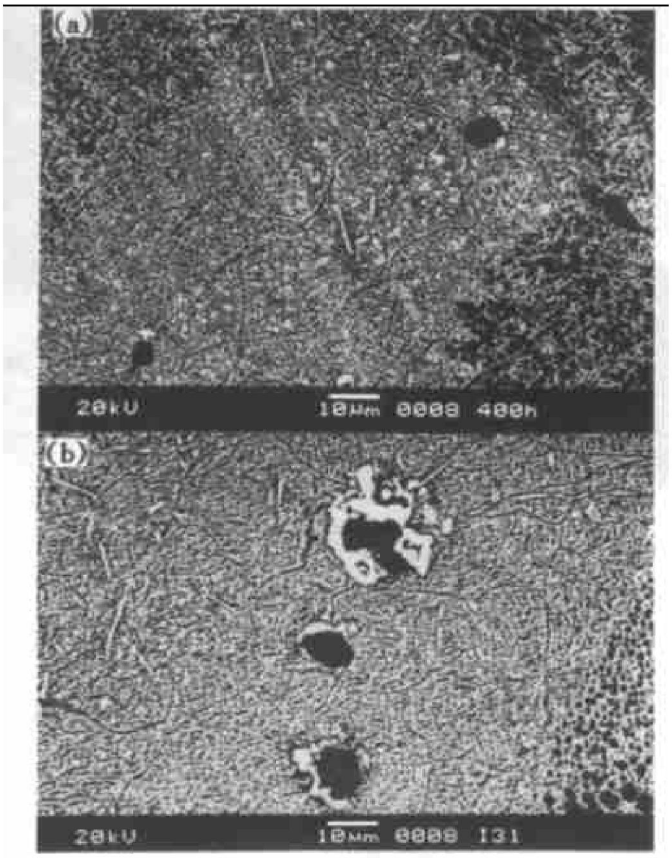

Fig. 5 BSEI of alloy IC6A after aging at 950 ℃
(a) —For 400 h; (b) —For 2 000 h

there is no obvious change in the region consisted of γ phase, Ni_3Y and $Mo_{1.24}Ni_{0.76}$ with the aging time up to 2 000 h. The size of γ and γ′ phases has no obvious change as well.

3.2 Effect of aging on mechanical properties

Fig. 6 gives the tensile properties at room temperature of alloy IC6A after aging at 950 ℃ for different time. From Fig. 6 it can be seen that the yield strength($\sigma_{0.2}$) decreases obviously for the alloy after aging for the first period of 500 h and has no further obvious change during further aging within 2 000 h. The ultimate tensile strength (σ_b) has no obvious change before aging for 1 000 h and decreases obviously during further aging . The decrement in ultimate tensile strength of alloy IC6A after aging for 1 000 h probably is mainly caused by the obvious increment of Y-NiMo phase in both size and amount.

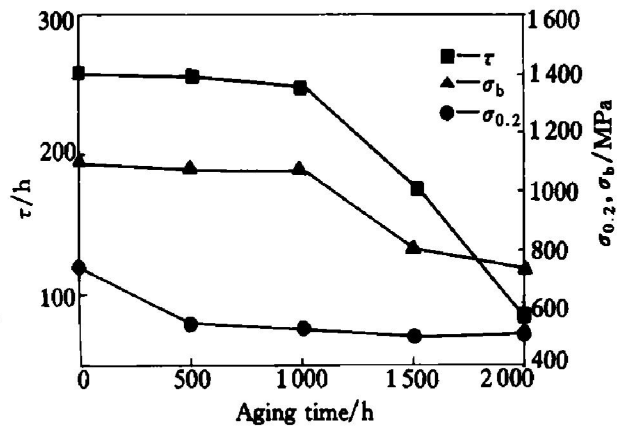

Fig. 6 Effect of aging time on mechanical properties of alloy IC6A

The effect of aging at 950 ℃ on high temperature stress rupture properties of alloy IC6A is also illustrated in Fig. 6. From the Larson-Miller curve of alloy IC6A (as shown in Fig. 7) , it can be inferred that the stress rupture life of alloy IC6A under 1 100 ℃, 90 MPa is about 257 h. The results in Fig. 6 show that the stress rupture life of alloy IC6A under 1 100 ℃, 90 MPa has no change for the specimens with aging time equal to or less than 1 000 h and decreases steadily for the specimens with aging time over 1 000 h. The decrement in stress rupture properties for the specimens after aging for 1 000 h is due to the increasing amount and coarsening of Y-NiMo phases.

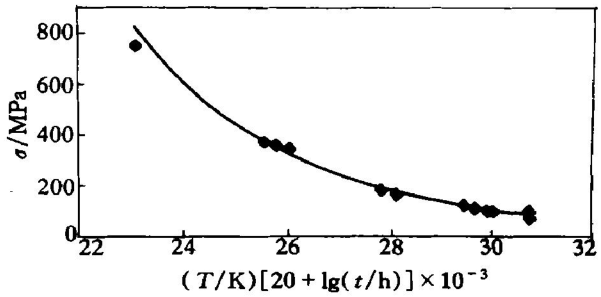

Fig. 7 Larson-Miller curve of alloy IC6A

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(Edited by YANG Bing)