RARE METALS
Vol. 30, Spec. Issue, Mar 2011, p. 345
DOI: 10.1007/s12598-011-0300-y

Effects of long-term aging at 1070 ºC on microstructure of Ni_3Al-base single-crystal alloy IC6SX

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Received 29 September 2010; received in revised form 29 December 2010; accepted 10 January 2011

Abstract

The effects of long-term aging at 1070 ºC for periods of 100 h to 1500 h on microstructure of Ni_3Al-base single-crystal alloy IC6SX were examined by SEM, EDX and TEM. Results show that alloy IC6SX undergoes following microstructure changes during long-term aging: a large number of rod-like Y-NiMo phases and carbides precipitate, and more of them appear in the interdendritic areas than in the dendritic arms; γ-phase aggregate and coarsen, and no longer show a continuous network-like distribution; adjacent γ' phases grow bigger and become connected with each other; some larger sized γ' phases are discovered in the dendritic arms; after aging for 300 h, γ phases amid a row of adjacent γ' phases dissolve to form a γ' band, and during further aging, γ phases beside the γ' band grow wider and finally occupy the γ' band, where a γ band is formed; a small portion of Y-NiMo phases are found to re-dissolve to form γ phases after aging for 1500 h.

Keywords: oxides; IC6SX; Ni_3Al; long-term aging; microstructure

1. Introduction

A single-crystal Ni_3Al-base high-temperature structural alloy, IC6SX, was developed from the unidirectional-solidified Ni_3Al-base alloy IC6 [1-2], by altering the contents of trace elements boron and carbon [3]. IC6SX is expected to be employed as components of aero-turbines, exposed to high temperature in long term, therefore the change in microstructure and mechanical properties of the alloy is of great importance to security and service life of those components.

Degradations of microstructure are quite common to high-temperature structural alloys after exposed to high temperature for a long time, which inevitably causes changes in mechanical properties [4-7]. Therefore, it is important to investigate the effect of long-term aging on high-temperature structural alloys [8].

Much research has been done about the long-term aging effects on microstructure of IC6 [9] and other Ni_3Al-base alloys [10-13]. It has been reported [9] that some microstructural changes occurred to directional-solidified alloy IC6 during long-term aging at 950 – 1150 ºC: the volume fraction of the main strengthening phase γ-Ni(Mo) solid solution decreased due to the formation of Y-NiMo phase by the reaction of γ→Y-NiMo+γ', both γ and γ' coalesced and grew.

2. Experimental

The material used for the present study was single-crystal alloy IC6SX, whose nominal chemical composition was Ni-(7.6-7.8)Al-14Mo-0.03B wt.%.

The single-crystal samples were obtained by unidirectional solidification by screw selecting method in a commercial induction furnace.

The as-cast samples underwent the standard heat treatment in air prior to long-term aging. The standard heat treatment was 1280 ºC, 10 h, air cooled and 870 ºC, 32 h, air cooled.

The long-term aging was preformed in a laboratory air muffle furnace at 1070 ºC for 100, 300, 600, 1000 and 1500 h.

The specimens were polished and etched for metallographic examination using a JEOLJSM-5600 scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS).

The thin foils were prepared by ion-beam thinning of specimens of 30 – 40 μm thick and then examined in a JEOL2000FX transmission electron microscope (TEM) for

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determination of the crystal structure of the precipitates.

3. Results and discussion

The dendritic nature of alloy IC6SX was still apparent after standard heat treatment, as shown in Fig. 1, where the darker parts represent the dendrite arms and the brighter ones indicated the interdendritic regions. Both the dendrite arms and the interdendritic regions appeared as a two-phase mixture of cubic γ' phases surrounded by net-like γ phases. The dimension of the cubic γ' phases was about 0.3 – 0.5 μm, and the thickness of the γ phases was about 0.1 μm.

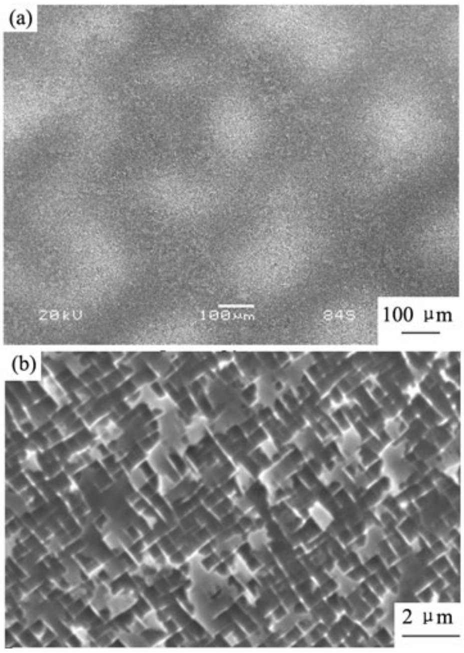

Fig. 1. Microstructure (SEI) of alloy IC6SX after standard heat treatment: (a) morphology of dendrites; (b) morphology of γ+γ'.

After aging at 1070 ºC for 100 h, some rod-like phases precipitated from the matrix, which displaced a homogeneous distribution. The rod-like phases had a length in the range of several to dozens of micrometers, and were brighter than γ and γ' phases in back scattered electron image (BSEI), as shown in Fig. 2, which indicated that they had a relatively high Mo content.

An obvious change in microstructure of IC6SX after aging for 300 h was that some large-sized γ' phases appeared in dendritic arms, and the dimension was about 10 – 30 μm. And the size of rod-like phases increased obviously, see Fig. 3.

The microstructural changes after aging at 1070 ºC for 600 h are shown in Fig. 4. The dimensions of the rod-like phases increased remarkably, and some of them were 10 – 20 μm long and 5 μm thick. An EDX analysis of the larger ones revealed their composition was 49 at.%Ni-50 at.%Mo-1 at.%Al. As more rod-like phases appeared, it was evident that they were more likely to precipitate in the interdendritic regions than in the dendrite arms, which was due to the reason that molybdenum, a main composition of the rod-like phases, was strongly enriched in the interdendritic regions.

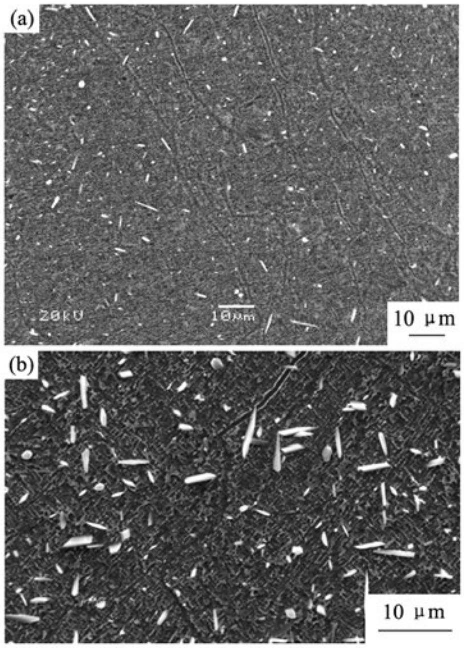

Fig. 2. Microstructure of alloy IC6SX after aging at 1070 ºC for 100 h: (a) SEI; (b) BSEI.

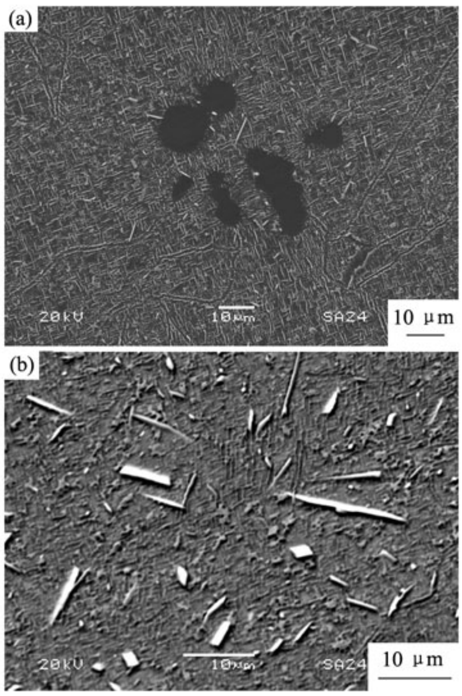

Fig. 3 Microstructure of alloy IC6SX after aging at 1070 ºC for 300 h: (a) large-sized γ' phases (SEI); (b) rod-like precipitates (BSEI).

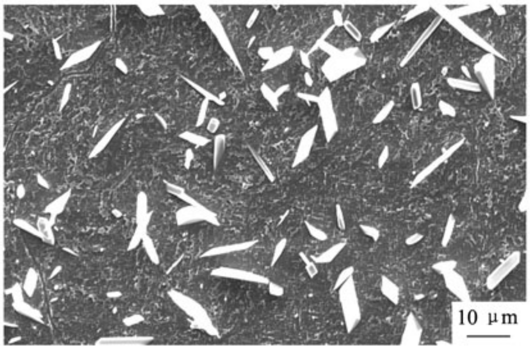

Fig. 4. Microstructure of alloy IC6SX after aging at 1070 ºC for 600 h.

Compared with previous studies[14-15] and based on TEM observations of specimens after aging for 600 h (see Fig. 5), some of the rod-like phases were carbides Mo_6Ni_6C with a face centered cubic structure, and others turned out to be Y-NiMo phases with base-centered orthorhombic structure,, which were formed by the reaction γ→Y-NiMo+γ' [14, 15].

γ bands of 1 – 3 µm wide were found after aging for 1000 h (Fig. 6 (c)), which could be traced back to a row of less regular-shaped γ' phases (Fig.6 (a)) after solution heat treatment (1280 ºC, 10 h, air cooled). After aging for 300 h (Fig. 6 (b)), the γ phases amid the row of γ' phases dissolved, and adjacent γ' phases were connected to form a γ' band. During further aging, the γ phases beside the γ' band grew wider and finally occupied the entire γ' band, where a γ band was formed (Fig. 6 (c)).

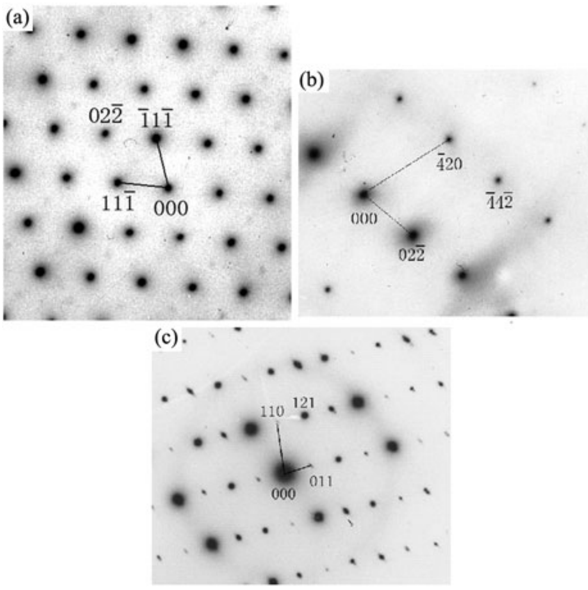

Fig. 5. TEM analysis of precipitates in IC6SX after aging at 1070 ºC for 600 h: (a) carbides Mo6Ni6C crystal zone axis [011]; (b) carbides Mo6Ni6C crystal zone axis [122]; (c) Y-NiMo phases crystal zone axis [1-11].

Some rod-like, about 10 µm-long γ phases appeared after aging for 1500 h (as marked by circles in Fig. 7), whose shape was similar to those precipitates formed during long-term aging. This was because of the reaction: Y-NiMo+γ'→ γ [15], the reverse progress of the formation of Y-NiMo, which indicated that Y-NiMo was a metastable phase, and the reaction products γ had a much lower Mo content than the previous γ [15].

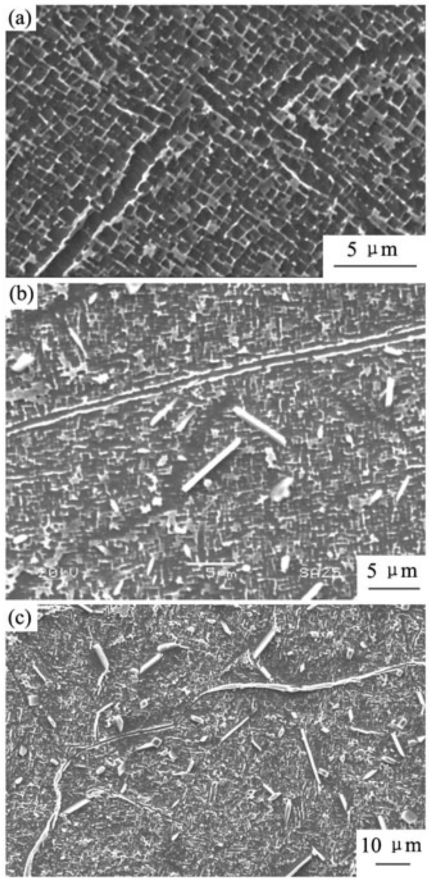

Fig. 6. Formation of γ band in IC6SX during aging at 1070 ºC (SEI): (a) after solution treatment; (b) aging period: 300 h; (c) aging period: 1000 h.

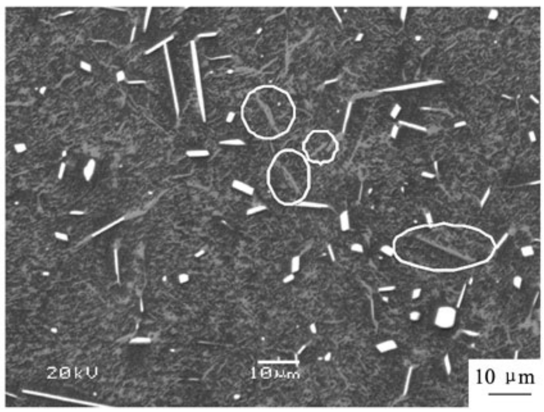

Fig. 7. Microstructure of IC6SX after aging at 1070 ºC for 1500 h (BSE) (rod-like γ phases marked by circles).

The microstructural change of the γ+γ' matrix was shown in Fig. 8. After aging for 100 h, some sections of the rod-like γ phases disappeared, and adjacent γ' became connected to each other. This trend continued after aging for 300 h. γ phases remarkably coarsened after aging for 1000 h, the thickness of which increased from about 0.1 μm before aging to 0.4 μm. After aging for 1500 h, most γ' became connected, γ phases isolated by γ'.

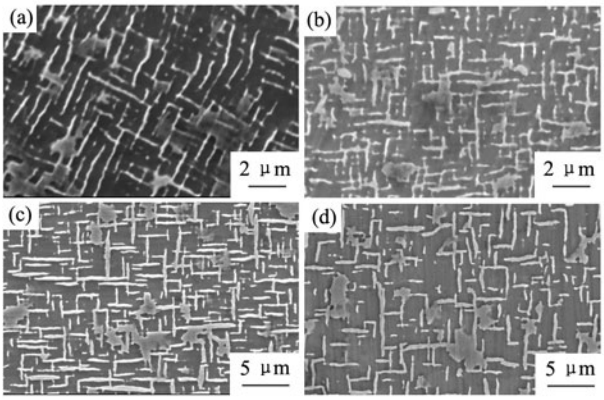

Fig. 8. Microstructural Change of γ+γ' in IC6SX during aging at 1070 ºC: (a) aging period: 100 h; (b) Aging period: 300 h; (c) aging period: 100 h; (d) aging period: 150 h.

4. Conclusion

After long-term aging at 1070 ºC, microstructure of alloy IC6SX underwent following changes: a large number of rod-like Y-NiMo phases and carbides Mo_6Ni_6C precipitated, and more of them appeared in the interdendritic areas than in the dendritic arms; γ-phase aggregated and coarsened, and no longer showed a continuous network-like distribution; adjacent γ' phases grew bigger and became connected with each other; some larger sized γ' phases were discovered in the dendritic arms; γ bands were formed after aging for 1000 h, which could be traced back to a row of less regular-shaped γ' phases; a small portion of Y-NiMo phases were found to re-dissolve to form γ phases after aging for 1500 h.

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