Diffusion induced recrystallization of Ni$_3$Al-based alloy

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Abstract. The paper represents the study of the kinetics of diffusion-induced recrystallization (DIR) and its effect on the disordering and creep resistance of an alloy based on Ni$_3$Al doped nickel aluminide. Recrystallization was observed in the Ni$_3$Al-based alloy hardened by the particles of chromium carbides at a temperature of 1573 K due to dissolution of carbides and diffusion of chromium in the volume of grains. Recrystallization leads to a sharp decrease in the creep resistance of the alloy due to the formation of an unordered $\gamma$ phase. The alloy can be used as a heat-resistant material at a temperature of not more than 1500 K.

1. Introduction
Ni$_3$Al-based alloys exhibit an abnormal temperature dependence of yield stress and high heat resistance, which allows us to consider these alloys to be promising heat-resistant materials. Phase and structural transformations in intermetallicides have some peculiarities [1]. These transformations at elevated temperatures, when alloys used as structural materials, can lead to a significant change in the mechanical properties. In this connection, the purpose of this work is to study phase transformations in the Ni$_3$Al-based alloy doped with chromium and carbon at elevated temperatures and the effect on creep resistance. Creep resistance tests were conducted in vacuum, using an upgraded PV-3012M machine [2] at 1373 K. The evolution of the phase composition and substructure during the diffusion-induced recrystallization was studied using a "Camebax microbeam" micro analyzer, a "Neofot-2" optical microscope and a JEM-F200 electron microscope.

2. Experimental results and discussion
In the initial state, after melding and further annealing at 1473 K for 4h, the alloy has an aggregate structure composed of equiaxed Ni$_3$Al grains with a size of $\sim 50 \mu$m and Cr$_7$C$_3$ particles with a volume fraction of $\sim 1.6\%$ located along the large-angle grain boundaries (figure 1a). The small-angle boundaries are free of particles. Further annealing at 1573 K leads to the dissolution of carbides and the development of DIR. DIR leads to the formation of a structure (figure 1b, c) that is morphologically similar to the structure of cellular decomposition of supersaturated solid solutions [3]. However, contrary to ordinary cellular decomposition, in this case, an increased concentration of the doping element (chromium) is observed not in the initial matrix, but in the transformed regions.

The formed structure of cells consists of the grains of the cubic $\gamma'$ phase 0.2-0.3 $\mu$m in size, between which the thin layers of the disordered $\gamma$ phase are observed (figure 1c). The cell is separated from the grain by a small-angle dislocation boundary. At the same time, the cell is separated from a neighbouring grain by the large-angle boundary. The initial grain boundaries do not move during the
growth of the cells. The boundaries of the cells have a diffuse dislocation structure (figure 1c), which indicates the presence of high diffusion stresses in the reaction front.

Hornbogen observed a similar reaction front during the formation of phases in iron-chromium alloys [4]. He proposed the mechanism for the growth of cells as follows. The formation of particles of a new phase leads to the occurrence of high stresses which are damped during the formation of dislocations. Incoherent particles are again formed on these dislocations due to the lower barrier of nucleation. Then the process develops auto catalytically.

The boundary of the grain is filled with chromium and carbon in the diffusion mode of C or B1 due to the dissolution of particles [6], when the maximum deviation from the equilibrium between the volume and the grain boundaries is observed. At the same time, high osmotic pressure occurs in grain boundaries, and high diffusion and concentration stresses appear in the boundary areas [6]. These stresses are damped during the formation of dislocations in the boundary area and further recrystallization. The boundaries of the recrystallized grains are again filled with a diffusant in the diffusion mode B1 and the process is repeated until the diffusion mode changes.

In the experiments the termination of DIR was observed before the carbides were completely dissolved, which was an indirect confirmation of this hypothesis.

Figure 2 shows the growth rate of the width of recrystallized zones as a function of time. The rate decreases faster $t^{-1/2}$, which may be associated either with depletion of diffusion sources or with a shift of the diffusion mode to the equilibrium one.

Figure 1. Structure of the Ni3Al alloy in the initial condition (a) and after DIR (b, c).

Figure 2. Growth rate of the recrystallized zone as a function of time. Curve 1 – experiment; curve 2 – the dependence $v\sim t^{-1/2}$.
To test the hypothesis concerning the realization of DIR in the diffusion mode C, the experiments were conducted for different heating rates of the samples. In ordinary experiments, the sample was heated at a rate of 50 K/min. When the heating rate was reduced to 2 K/min in the range from 1273 to 1473 K, DIR was not observed. We assume that in this case the filling of the grain boundary with the dissolved elements is controlled not by diffusion, but by the dissolution rate of the particles. In this case, the volume of grains is saturated with chromium due to volume diffusion, which is equivalent to the shift of the diffusion mode to more equilibrium B$_2$ and B$_3$.

The size of the new grains (0.2-0.3 μm) correlates with the width of the zone with an increased density of dislocations. The front propagation of DIR is discrete. For some time, the front is in the "waiting" state and then quickly propagates over a distance of 0.2-0.3 μm. In the "waiting" state, there is an increase in the width of the zone with a high dislocation density before the reaction front. The initial equiaxial grain structure cannot be restored after DIR only using heat treatment.

As noted above, Ni$_3$Al-based alloys are of interest as structural heat-resistant materials. So, the effect of DIR on creep was studied.

The alloy is fractured in the case when the viscous intercrystalline mechanism of creep is observed (figure 3).

![Figure 3. Structure of the Ni$_3$Al alloy after creep (ε=10%).](image)

![Figure 4. Creep curves of the Ni$_3$Al alloy in the initial state (1) and after DIR (2).](image)
Cracks are formed in the boundary intersection of the grains and grow along the grain boundaries which contain carbide particles and make an angle with the applied stress close to \( \pi/2 \). The growth of individual intercrystalline cracks is controlled by grain-boundary diffusion [8]. The coalescence of individual cracks occurs at the third stage of creep along unfavourably oriented grain boundaries and along the body of grains. This process is much slower compared with the growth of individual cracks. Therefore, the duration of the third stage of creep turns out to be comparable with that of the second stage of creep (figure 4).

Creep tests were conducted at 1373 K under load of 40 MPa. The one batch of samples had the initial structure, and the second batch of samples was preliminarily subjected to DIR at 1573 K. As a result, the plasticity and creep resistance of the alloy are sharply reduced after DIR. The fracture was viscous and occurred along recrystallized areas.

3. Conclusion

Recrystallization was observed in the Ni\textsubscript{3}Al-based alloy hardened by the particles of chromium carbides at a temperature of 1573 K due to dissolution of carbides and diffusion of chromium in the volume of grains. The formed structure of the cells consists of the grains of the cubic \( \gamma' \) phase 0.2-0.3 \( \mu \)m in size, between which thin layers of the disordered \( \gamma \) phase are observed. The reason of recrystallization, apparently, is the diffusion mode \( B_1 \) taking place during the diffusion filling of the grain boundaries due to the fast dissolution of carbide particles. This is confirmed by the suppression of DIR during slow heating, when the saturation of the boundaries and volume of grains is controlled by the dissolution rate of particles. The boundaries and volume of grains in this case are in a more equilibrium state compared to that in the diffusion modes C and \( B_1 \). In addition, doping of Ni\textsubscript{3}Al with chromium reduces the temperature of its disordering.

Recrystallization leads to a sharp decrease in the creep resistance of the alloy due to the formation of an unordered \( \gamma \) phase. The alloy can be used as a heat-resistant material at a temperature of not more than 1500 K.

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