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Influence of long-range ordering on mechanical properties of nanocrystalline Ni_3Al

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Abstract

The influence of a grain size and long-range ordering on mechanical properties of the intermetallic compound Ni_3Al at room temperature was investigated. It has been found that the maximum values of strength and ductility are observed in the material in a completely disordered nanocrystalline state. © 2001 Acta Materialia Inc. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Intermetallic compound Ni_3Al; Plastic deformation; Fracture

Introduction

The intermetallic compound Ni_3Al exhibits abnormal temperature dependence of flow stress. Its flow stress increases with temperature [1–3]. Another important feature of this material is grain boundary brittleness at room temperature leading to premature fracture [4]. Ductility of the material can be improved by additional microalloying, for example via boron addition [5–7]. Another efficient method of improving mechanical properties of the intermetallic compound Ni_3Al is structure refinement [8,9]. One of the method offering such refinement, down to submicrocrystalline (SMC) and nanocrystalline (NC) size, is severe plastic deformation (SPD) [8]. In the previous works [9–13] it was shown that the NC structure of Ni_3Al can be obtained via SPD in a completely disordered state and in a state with partial long-range ordering. These findings open a question of relative influence of grain refinement and long-range ordering on mechanical properties. In the literature no data has been found about the influence of a

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long-range ordering degree on mechanical properties. This subject is therefore the aim of the present work.

Experimental

The Ni_3Al (25.0 at.%Al, 75 at.%Ni) was prepared by melting high purity Ni and Al in an inductive plasma furnace. The alloy was homogenised by annealing for 40 h at 1323 K under a vacuum of 10^{-6} Pa. The specimens, 6 mm in diameter and 0.3 mm thick, were subjected to shear deformation by torsion at room temperature under a quasi-hydrostatic pressure of 8 GPa in a Bridgman anvil-type unit. After such treatment, we obtained specimens about 8 mm in diameter and 0.15 mm thick. The total strain of the specimens can be estimated as approaching 200 [14]. The deformed material was annealed in the temperatures ranging from 293 to 1313 K for 40 min in a vacuum of 10^{-6} Pa. The transmission electron microscopy (TEM) studies of the foils were conducted on a JEM-2000 FX transmission electron microscope. The foils were prepared by jet polishing. The fracture surfaces of the failed samples were examined in a scanning electron microscope JSM-840.

The long-range ordering parameter (LRO) was estimated by X-ray structural analysis. The LRO values were determined from the ratio between the intensities of the $\{1\,1\,0\}$ superlattice line and the $\{2\,2\,0\}$ fundamental line. This ratio was corrected by using the intensities of ordered Ni_3Al samples obtained by annealing and slow cooling. Methods of measurement of LRO and internal strains were described earlier [13].

Microhardness was measured using a Vickers indenter with a load of 0.2 kg applied for 15 s. The three-point bending tests were conducted on a testing machine IR5057-50 equipped with a gauge having a maximum load of 50 N. The specimens for bend testing were cut out on an electric spark machine in distilled water and finished to the final dimensions of 0.15 mm × 1.5 mm × 8 mm by polishing and grinding. The bending stresses were calculated from the following equation: $\sigma = 1.5PL/(bh^2)$, where P is the load, L is the distance between supports, h is the height and b is the width of the specimen [15].

Strain of the specimen in three-point bending test is non-uniform and depends on the distance from neutral axis and reaches a maximum on the outer surface of the specimen. For a small deformation there is proportional relationship between strain and radius of curvature of the specimen. Maximum plastic deformation at failure was used to characterise the ductility of specimens.

Results

The TEM studies of the as-deformed state of Ni_3Al showed that the alloy had a NC structure (Fig. 1).

The main specific feature of such a structure is the presence of fuzzy grain boundaries on which complex diffraction contrast is observed. The mean grain size estimated on

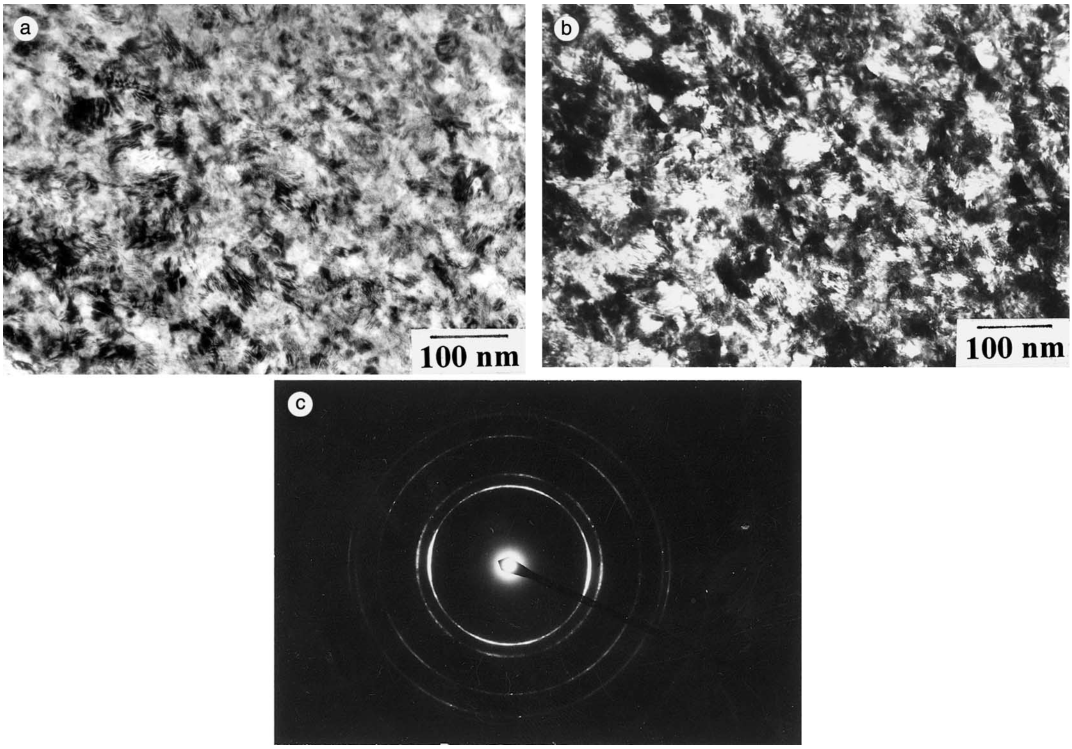

Fig. 1. Structure of the intermetallic compound Ni_3Al subjected to severe plastic deformation (SPD) in a Bridgman anvil-type unit: (a) bright field image, (b) dark field image, (c) diffraction pattern from an area of 0.5 μm^2.

dark field images is about 20 nm. The diffraction pattern with a large number of spots arranged in circles provides evidence for the existence of large misorientations between nanocrystallites. Thin light and dark bands of alternating contrast are observed inside of some grains. The X-ray structural analysis of the as-deformed state showed that the alloy was entirely disordered (LRO = 0) and was characterised by a high value of internal strains (Fig. 2).

The microhardness of samples processed by SPD was 6200 MPa, the bending strength was 2800 MPa and maximum plastic strain ε_{pl} was 1.44%. Annealing of deformed specimens in the range of temperatures up to 420 K does not change the long-range order, grain size and internal strains. As a result of annealing at 533 K, a significant increase of LRO parameter up to $\approx$0.3 was observed. At the same time, the values of the internal strains decreased to 0.2%, without any significant growth of the coherent diffraction domains. The TEM observations show that the crystallites become clearer, the structure of the boundaries is somewhat improved and a slight enlargement of some grains is observed. However, significant changes of mechanical properties take place: ductility drops to zero and the ultimate bending strength decreases to 1350 MPa. These changes are accompanied by an increase of microhardness to 9200 MPa (Fig. 3).

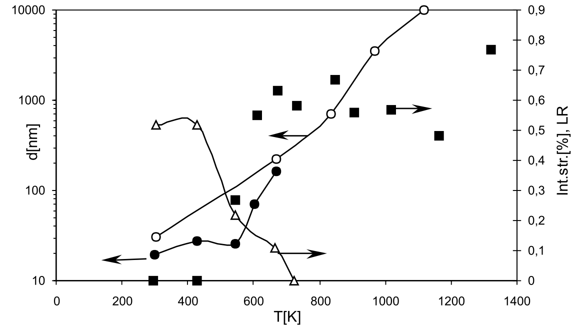

Fig. 2. Dependence of structure parameters on temperature of annealing for the intermetallic compound Ni_3Al subjected to SPD: (○) TEM data of grain size d (nm), (●) X-ray structural analysis data of grain size d (nm), (■) long-range order parameter LRO, (△) level of internal strains (%).

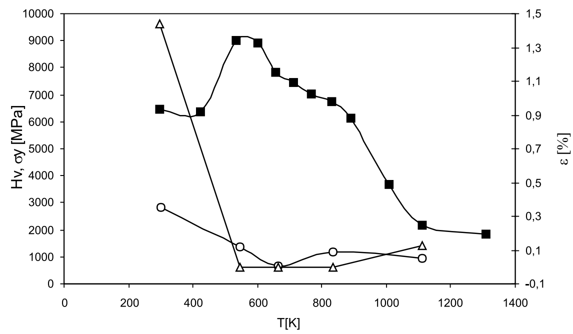

Fig. 3. Dependence of mechanical properties on temperature of annealing for the intermetallic compound Ni_3Al subjected to SPD: (■) Vickers microhardness H_v (MPa), (△) plastic deformation at failure ε (%), (○) ultimate bending strength σ_y (MPa).

Due to annealing at 600 K, the LRO parameter reaches a value about 0.6. Internal strains continue to decrease and some amount of grain growth occurs. Microhardness remains high and elongation remains zero, with some decrease of ultimate tensile stress. At 900 K the internal strains decrease to zero, the mean crystallite size grows to about 3 μm. Microhardness decreases slowly to reach about the value of prior to plastic deformation. There is still no visible plasticity, the ultimate bending strength value reaches a minimum around 650 K.

By annealing at 1113 K, grain size increases sharply and reaches 10 μm (Fig. 2). Twins and antiphase boundaries may be seen inside the grains. Microhardness decreases to 2000 MPa. A striking feature of the deformation behaviour is that some amount of plasticity is recovered.

Fig. 4 shows the fracture surfaces after bending tests at room temperature. Examination of the fracture surface of the as-deformed samples revealed both intergranular and transgranular fracture modes (Fig. 4a). The fracture mode of all the annealed

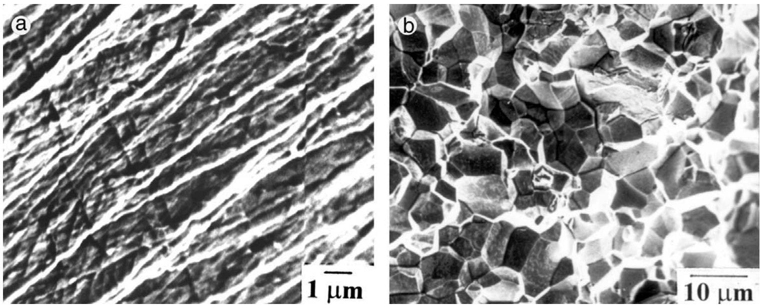

Fig. 4. Fracture surfaces after bending tests at room temperature: (a) as deformed specimen, (b) specimen annealed at 1113 K.

samples was intergranular one. An illustration is a fracture surface of the sample annealed at 1113 K (Fig. 4b).

Discussion

The influence of structure on the deformation behaviour and mechanical properties of the present intermetallic compounds should be considered as resulting from a competition between plastic deformation and fracture. Their relative importance is determined by a number of factors. In principle, both ordering and grain refinement are physically independent mechanisms strengthening polycrystals. From that point of view the highest strength in the present case should be observed in the specimen annealed in the range of temperatures from 600 to 800 K. However, Ni_3Al exhibits relatively low fracture stress and specimens characterised by ordered structure and fine grain size break before reaching yield point. In fact the fracture stress of fine-grain size ordered specimens practically does not depend on annealing temperature. In this situation grain growth, which decrease flow stress and hardness brings about a measurable ductility of tensile strained specimens.

Another point to be noted is that the as-deformed material was completely disordered and as such material cannot be strictly considered an intermetallic compound, but rather solid solution hardened by a high degree of cold work. The significant ductility of this structure is related to the absence of the usual, well-defined grain boundaries. As deformed state has fuzzy, poorly defined grain boundaries and in turn absence of grain boundary stress concentrations, this corresponds to measurable plastic deformation before fracture.

The sharp decrease in ductility observed due to annealing at 533 K takes place with a partial long-range ordering. This ordering contributes to brittleness, even if it concerns only a fraction of the sample volume. Such a phenomenon can be explained as follows. First, the areas with an ordered structure should have higher values of the elastic modulus and therefore generate higher stresses at a given imposed strain. As a result the fracture stress might locally exceed the critical level in the ordered region. Secondly, this could lead to a in-homogeneity of deformation. The local deformation – ε^l (deformation within small volumes comparable with the dimensions of Griffith cracks) can be many times higher than the mean deformation of specimen – ε_m. A high degree of in-homogeneity ($\varepsilon^l/\varepsilon_m > 100$) should lead to a crack growth by a brittle mechanism [16].

The microhardness variation (which increases at annealing at 533 K) might appear to contribute to the observed decrease of the ultimate strain. However the microhardness measurements actually indicate an increase of the flow stress of the material. By contrast, the ultimate bending stress reflects the stress level at which fracture occurs, and can be significantly lower than the fracture stress during the elastic loading.

Finally, an interesting observation is the recovery of some ductility by annealing at 1113 K. This occurs simultaneously with a large increase of grain size (from about 0.7 μm at 833 K where the material is brittle, to about 10 μm at 1113 K). It may be supposed that a brittle behaviour should again be observed at still larger grain size, since pure undeformed annealed Ni_3Al exhibits grain boundary brittleness. The occurrence of an optimum grain size with respect to ductility may be compared with observations performed in TiAl with a SMC structure obtained by thermomechanical treatment [17]. The TiAl sample with a grain size of $d = 8$ μm had the maximum ductility at room temperature and both a decrease and an increase in grain size resulted in a decrease in ductility. The authors [17] considered that a favourable combination of deformation mechanisms, namely intragranular dislocation slip and twinning, are responsible for this effect.

Conclusions

1. The structure and mechanical properties of the intermetallic compound Ni_3Al subjected to SPD in a Bridgman anvil-type unit and to subsequent annealing were investigated.
2. It was shown that due to SPD an entirely disordered structure with a grain size of 20 nm was formed in the material. A non-monotonic dependence of hardness, flow stress and ductility (at 20°C) on the temperature of annealing was observed.
3. The material has maximum strength and ductility in the as-deformed disordered NC state. Annealing of deformed specimens increases long-range order and in the range of nano and SMC grain sizes leads to a complete loss of ductility and decrease in strength. A subsequent increase in ductility and strength was observed when the grain size increased to 10 μm.
4. The influence of a grain size and long-range ordering on the mechanical properties of Ni_3Al reflects an interplay between the processes of plastic deformation and fracture.
5. The results obtained in present study suggest that grain boundaries in the NC Ni_3Al undergo transformation during low temperature annealing. This effect, however, requires further confirmation.

Acknowledgements

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