Superplasticity in a Recrystallized Ni₃Al Polycrystal Doped with Boron

By M. S. Kim*, S. Hanada**, S. Watanabe** and O. Izumi**

Superplasticity in a recrystallized Ni₃Al polycrystal doped with boron was investigated. Superplastic flow with a maximum elongation of 160% was observed under the condition of an ultra fine grain size (1.6 μm) at a low initial strain rate ($\leq 1.04 \times 10^{-4} \text{ s}^{-1}$) and 973 K. A low flow stress and a high strain-rate sensitivity index were attained under these conditions.

The occurrence of a dynamic recrystallization (DR) was verified by a transmission electron microscope (TEM), and DR was believed to be an important mechanism in superplastic deformation as associated with a reduced cavitation. This limited cavitation was explained by considering that DR relieves stress concentrations arising from grain boundary sliding and dislocation piling-up at grain boundaries. In addition, DR played another important role in suppressing apparent grain growth during superplastic deformation.

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I. Introduction

Ductilized Ni₃Al-based compounds are considered to be promising as a practical high temperature material, since these compounds have excellent high temperature properties; a positive temperature dependence of flow stress, superior creep-, fatigue-, corrosion-resistance and relatively low density.

Improvement in ductility of single-phased Ni₃Al has been made at room temperature by doping a small amount of boron in recent years⁽¹⁾, and at elevated temperatures by refining a grain size later⁽²⁾⁽³⁾. Especially, we have found⁽³⁾ superplasticity in a recrystallized Ni₃Al polycrystal doped with boron having an ultra fine grain size of 1.6 μm and a single phase of L1₂ ordered structure. Superplasticity, the ability to deform extensively under low stresses without necking, has been observed in a wide variety of metallic alloys⁽⁴⁾ and certain ceramics⁽⁵⁾⁽⁶⁾. Nevertheless, superplastic

behavior in a single-phased intermetallic compound has not been reported except only a few cases⁽⁷⁾⁻⁽⁹⁾. This fact is probably resulting from a limited investigation on tensile properties owing to a preconceived idea that intermetallic compounds are brittle. It has been predicted that at elevated temperatures the mechanical properties of a boron-doped Ni₃Al polycrystal are strongly dependent of a grain size, test temperature and strain rate. Indeed, careful control of these variables has resulted in the superplastic behavior with the maximum elongation of 160%⁽³⁾.

Interest in superplasticity of Ni₃Al may extend rapidly with the realization that superplastic materials have a wide potential application in many industrial forming operations with cost reductions and considerable freedom in designing complex components. At present, there are a considerable number of papers describing experimental and theoretical aspects of superplastic deformation in metallic alloys, while no single model has had universal acceptance.

It is the purpose of this paper to clarify the mechanism and condition of superplasticity in single phased intermetallic compound Ni₃Al doped with boron. We present the results on

* Graduate Student, Department of Materials Science and Engineering, Tohoku University, Sendai 980, Japan.

** Institute for Materials Research, Tohoku University, Sendai 980, Japan.

the relation between strain rate, grain size, temperature, superplastic deformation parameters and flow stress, together with the deformation characteristics of the alloy. In particular, detailed studies of cavitation have been carried out and this work has been focused on the metallographic features of flow and fracture.

II. Experimental

Ni₃Al (24 mol% Al) alloys containing 0.24 mol% B were prepared by arc-melting and homogenized as described elsewhere⁽¹⁰⁾. Rectangular tensile specimens having gage section of 1 mm × 2 mm × 16 mm were spark-machined from the cold rolled plates. Recrystallization annealing at various temperatures was performed to obtain the requisite grain sizes of 1.6, 2.9, 8.5 and 28 μm . The grain size was confirmed by the linear intercept method using both an optical (OM) and transmission electron microscope (TEM). The microscopic observations also revealed that the specimens had equiaxed grains with uniform distribution of grain size. Thin foils for TEM studies were produced by jet electropolishing.

After the surface layer of the tensile specimens was removed by electropolishing, tensile tests were conducted on an Instron-type testing machine at an initial strain rate from 5.2×10^{-5} to $2.08 \times 10^{-3} \text{ s}^{-1}$ and at temperatures 673 to 973 K in a vacuum of $2 \times 10^{-3} \text{ Pa}$. During tensile testing the samples and their grips were loosely wrapped with a Ti foil to keep the surface clean.

The longitudinal section of specimens was observed by both an OM and a scanning electron microscope (SEM). SEM observations were also made on the fracture surface. Furthermore, the internal structure of specimens deformed to 0.21 true strain was examined by TEM.

III. Results

Figure 1 shows the engineering stress-strain curves for specimens having the grain size of 1.6 μm at an initial strain rate ($\dot{\epsilon}$) of 5.2×10^{-5} to $2.08 \times 10^{-3} \text{ s}^{-1}$ and at 973 K. There is an initial rapid rise in stress to a maximum, follow-

Fig. 1 Engineering stress-strain curves for the specimen having a grain size of 1.6 μm deformed at 973 K with various initial strain rates.

Fig. 2 True stress-true strain curves for the superplastic specimen, converted from Fig. 1.

ed by a rapid reduction in stress ($\dot{\epsilon} \geq 2.08 \times 10^{-4} \text{ s}^{-1}$) or by a relatively steady one ($\dot{\epsilon} \leq 1.04 \times 10^{-4} \text{ s}^{-1}$). The rapid drop in stress corresponds to the development of necking in addition to the overall reduction in specimen cross-section with deformation. In contrast, the steady reduction in stress results from the latter effect, because necking occurred only at the final stage of deformation. Therefore, the conversion to true stress-true strain curves can be obtained in the conventional way and the results are shown in Fig. 2. Figure 2 shows clearly steady-state deformation to large strain without necking, which is characteristic of superplastic behavior⁽¹¹⁾⁻⁽¹⁵⁾.

Fig. 3 TEM micrograph illustrating the internal structure of the specimen, grain size (d) $1.6\text{ }\mu\text{m}$ deformed superplastically to 0.21 true strain at 973 K and initial strain rate ($\dot{\epsilon}$) of $5.2 \times 10^{-5}\text{ s}^{-1}$. Note the recrystallized grains containing dislocations.

A typical TEM micrograph depicting the internal structure of a specimen deformed superplastically to 0.21 true strain is given in Fig. 3, where the specimen is pulled with the initial strain rate of $5.2 \times 10^{-5}\text{ s}^{-1}$. It is evident that all the grains are equiaxed and well developed dislocations with a high density are observed in almost all the grains. No apparent dislocation cell structure is found. Therefore, at least some of the grains are considered to result from DR, although we cannot distinguish DR grains from original ones. Similar observation of DR by TEM has been done in boron-free Ni₃Al⁽¹⁶⁾.

Representative OM micrographs of the polished longitudinal section at fracture after superplastic deformation ($\dot{\epsilon} = 5.2 \times 10^{-5}\text{ s}^{-1}$) and conventional one ($\dot{\epsilon} = 5.2 \times 10^{-4}\text{ s}^{-1}$) are shown in Fig. 4(a) and (b), respectively. Note that the superplastically deformed specimen contains a considerably lower density of cavities than conventionally deformed one does. To measure the grain morphology of the

Fig. 4 Optical micrographs of the polished longitudinal section of fractured specimen; (a) $d = 1.6\text{ }\mu\text{m}$, $\dot{\epsilon} = 5.2 \times 10^{-5}\text{ s}^{-1}$, 973 K, (b) $d = 1.6\text{ }\mu\text{m}$, $\dot{\epsilon} = 5.2 \times 10^{-4}\text{ s}^{-1}$, 973 K, (c) $d = 1.6\text{ }\mu\text{m}$, $\dot{\epsilon} = 5.2 \times 10^{-5}\text{ s}^{-1}$, 873 K, (d) $d = 2.9\text{ }\mu\text{m}$, $\dot{\epsilon} = 5.2 \times 10^{-5}\text{ s}^{-1}$, 973 K.

specimen deformed superplastically, a light etching was done in the longitudinal section using the same specimen as Fig. 4(a). The result is shown in Fig. 5, indicating an equiaxed grain structure and no evidence of grain elongation. In addition, limited grain growth is observed. The grain size does not increase more than double the initial grain size.

Figure 6 shows SEM micrographs of the fracture surface of the superplastic specimen (Fig. 6(a), $\dot{\epsilon} = 5.2 \times 10^{-5} \text{ s}^{-1}$) and the conventional one (Fig. 6(b), $\dot{\epsilon} = 2.08 \times 10^{-3} \text{ s}^{-1}$). Relatively large cavities are visible in the fracture surface of the superplastic specimen. This fact indicates that significant cavitation probably arises at large strains in the superplastic specimens. In contrast, for conventionally deformed specimens, a large amount of cavitation initiated at smaller strain with occurrence of necking. This is shown in Fig. 4(b), indicating increased volume fraction of cavities in the longitudinal section.

Figure 7 shows the initial strain rate dependence of the elongation (strain to fracture), the peak stress (σ : maximum stress at an initial stage of deformation, obtained from a true stress-true strain curve) and the strain rate sensitivity index (m). Similarly to most superplastic materials, m is defined as $m =$

Fig. 5 SEM micrograph of the etched area of Fig. 4(a). Note the equiaxed grains after superplastic deformation.

Fig. 6 SEM fractographs of the specimen, grain size $1.6 \mu\text{m}$ deformed at 973 K (a) superplastically ($\dot{\epsilon} = 5.2 \times 10^{-5} \text{ s}^{-1}$) and (b) conventionally ($\dot{\epsilon} = 2.08 \times 10^{-3} \text{ s}^{-1}$).

$\delta \log \sigma / \delta \log \dot{\epsilon}$ resulting from the equation $\sigma = K \dot{\epsilon}^m$, where K is a material constant for a given testing condition. It is well established that m value is an important parameter in superplasticity. It describes the capacity of the material to resist necking⁽¹⁷⁾⁻⁽¹⁹⁾. In general, m value depends on $\dot{\epsilon}$, grain size (d) and test temperature. A superplastic flow occurs when m is greater than 0.3 ⁽¹³⁾⁽²⁰⁾⁻⁽²⁴⁾ in the limit of $m = 1$, Newtonian viscous flow. As can be seen from Fig. 7, the m value increases with decreasing $\dot{\epsilon}$ and when m becomes greater than 0.3 ($\dot{\epsilon} < 2.08 \times 10^{-4} \text{ s}^{-1}$), a superplastic elongation over 150% is obtained with a neck-free flow as mentioned above.

Fig. 7 Variations of the elongation, flow stress (σ) and strain rate sensitivity index (m) with initial strain rate at 973 K for the specimens having a grain size of 1.6 μm .

It has been clearly demonstrated that the occurrence of superplasticity is depending on grain size and test temperature in addition to $\dot{\epsilon}$. The influence of grain size on the engineering stress-strain curve is illustrated in Fig. 8 for specimens tested at 973 K with $\dot{\epsilon}$ of $5.2 \times 10^{-5} \text{ s}^{-1}$. The specimens having grain sizes of not smaller than 2.9 μm exhibit abrupt decrease in the stress with increasing strain after reaching a peak stress, while the specimen with a grain size of 1.6 μm shows superplasticity. Figure 9 shows $\dot{\epsilon}$ dependence of elongation and flow stress as a function of grain size. From the slope of the log σ vs log $\dot{\epsilon}$ curve in Fig. 9, the specimens with a larger grain size than 1.6 μm are found to have a lower m value than 0.3. Both decreased elongation and increased flow stress with increasing grain size at a given $\dot{\epsilon}$ are also seen in Fig. 9.

SEM examination revealed that the grain boundary offsets and wedge-type cracks, which are evidence of grain boundary sliding (GBS), were frequently observed after deformation in

Fig. 8 Engineering stress-strain curves for different grain sizes at 973 K and $\dot{\epsilon} = 5.2 \times 10^{-5} \text{ s}^{-1}$.

Fig. 9 The initial strain rate dependence of the elongation and flow stress (σ) as a function of grain size.

the longitudinal section of coarse-grained specimens pulled with $\dot{\epsilon}$ of $5.2 \times 10^{-5} \text{ s}^{-1}$. This observation supports the previous conclusion that GBS occurs above 673 K in a boron-doped Ni₃Al⁽²⁵⁾⁽²⁶⁾.

SEM fractographs of coarse-grained specimens with $\dot{\epsilon}$ of $5.2 \times 10^{-5} \text{ s}^{-1}$ showed that the fracture mode changed with grain size. Namely, the specimens with $d \geq 8.5 \mu\text{m}$ exhibited intergranular fracture with a dimple-

like pattern, while deep cavities were observed in $d=2.9\text{ }\mu\text{m}$. The latter observation seems to correspond to Fig. 4(d), indicating a large volume fraction of cavities in the polished longitudinal section.

The effect of test temperature on an engineering stress-strain curve is indicated in Fig. 10, for specimens having a grain size of $1.6\text{ }\mu\text{m}$ and strained at $\dot{\epsilon}$ of $5.2 \times 10^{-5}\text{ s}^{-1}$. The stress-strain curve at 873 K exhibits remarkable decrease in flow stress, and fracture surface consisted of a large number of deep cavities. In contrast, the specimen tested at 673 K shows marked work hardening and cup and cone typed fracture appearance. The cup and cone typed fracture has also been observed at 290 K in the same composition of alloys having a fine grain size ($d < 8.5\text{ }\mu\text{m}$)⁽¹⁰⁾. It is widely accepted that the cup and cone typed fracture begins as a normal rupture when cavities (or voids) are formed and coalesced by dislocation interactions at a high stress level enough to open the cavities. It is interesting to note that the fracture at elevated temperatures is also controlled by the cavitation while the cup and cone typed appearance is not developed. This fact will be discussed in the next section of this paper. Shortly speaking, the cavities which are the basic source of the fracture, are formed primarily by GBS at elevated temperatures, while by dislocation interaction at low tempera-

Fig. 10 Engineering stress-strain curves for the specimens having a grain size of $1.6\text{ }\mu\text{m}$ deformed at $\dot{\epsilon}=5.2 \times 10^{-5}\text{ s}^{-1}$ with various test temperatures.

Fig. 11 The initial strain rate dependence of the elongation and flow stress (σ) as a function of test temperature.

tures.

An OM micrograph in the polished longitudinal section of the specimen deformed at 873 K is exhibited in Fig. 4(c), showing an increased volume fraction of cavities comparing with that of the superplastic specimen tested at 973 K.

Figure 11 shows the $\dot{\epsilon}$ dependence of elongation and maximum flow stress as a function of test temperature. Note that decreasing temperature leads to both decrease in elongation and increase in flow stress at a given $\dot{\epsilon}$. Furthermore, m value becomes lower than 0.3 with decreasing temperature.

IV. Discussion

Superplastic deformation in a boron-doped Ni_3Al polycrystal seems to be related to dynamic recrystallization, grain boundary sliding and a contribution to the total strain from slip. Now we discuss which process is the prime controlling factor for superplasticity in this compound based on the present microscopic observations. If grain boundary

sliding plays an important role, microstructure during superplastic deformation would consist of dislocation-free grains as confirmed in many superplastic alloys. Next, if crystallographic slip controls superplastic deformation, elongated grains would be developed in the microstructure. However, these postulates are in conflict with the experimental observation that the grain structure remains equiaxed during superplastic extension and most of grains contain a high density of dislocations. Therefore, dynamic recrystallization appears to be an important process operating during superplastic deformation in this compound. Namely, the alternative models proposed to explain superplasticity in metallic alloys, such as grain boundary sliding^{(27)–(31)}, crystallographic slip⁽³²⁾⁽³³⁾ or diffusional creep^{(34)–(36)} do not hold for a boron-doped Ni₃Al polycrystal.

It has been revealed in present investigation that important requirements for superplastic deformation of a boron-doped Ni₃Al polycrystal are as follows: 1) a fine equiaxed grain size of 1.6 μm , 2) a critical temperature of 973 K which is approximately half the melting point of Ni₃Al⁽³⁷⁾, 3) a high m value larger than 0.3 and 4) an initial strain rate not larger than $1.04 \times 10^{-4} \text{ s}^{-1}$. Similar requirements are also known to exist in other superplastic materials. To understand experimental results listed above, we will discuss the formation and growth of cavities, because the suppression of cavitation is directly related to large superplastic elongation as shown in Fig. 4.

It is well established that cavity formation arises under the combined action of GBS and flow stress at temperatures where GBS occurs. The general model of cavitation is based on the idea that stress concentration at grain boundaries, which is accompanied with GBS, results in the decohesion of the boundary interface. Such concentrated stresses could be relieved by some accommodation process, such as DR. If the competition between the opening of a cavity by GBS and its closure (accommodation) by DR is considered, the cavity will hardly be formed when the latter predominates over the former. This has been verified by the present microstructural observation that cav-

itation is suppressed as DR is accelerated in the superplastic specimens. Therefore, DR is presumably an important mechanism for superplasticity in a boron-doped Ni₃Al polycrystal.

As mentioned above, the cavity formation is also influenced by flow stress. As shown in Figs. 7, 9 and 11, superplastically deformed specimens have a considerably low value of flow stress. This fact implies that the tendency for void formation decreases with decreasing stress level.

When cavities are formed in the specimen, failure occurs by the growth and coalescence of these cavities. In superplastic specimens, significant cavity growth begins probably at higher strain, because necking is produced at almost final stage of deformation. It is now well demonstrated that the high value of m confers a resistance to neck growth. Under the condition of a high m value, which is greater than about 0.3 for most superplastic materials, the necks are diffuse and do not propagate, and the areas of reduced cross-section deform at similar rates to the remainder of the specimen, thus the behavior is superplastic. In practice, examination of Figs. 7, 9 and 11 shows that elongation to fracture increases with increasing value of m and superplastic flow occurs when $m > 0.3$.

In addition to the m value, the influence of both flow stress and GBS on cavity growth should be taken into account, because a cavity grows under an applied stress and is also controlled by the extent of GBS. In a similar manner to the void formation described above, both a low applied stress and occurrence of DR by which GBS is relaxed, should hinder a cavity growth. Moreover, DR retards apparent grain growth and leads to superplastic deformation. Therefore, it can be concluded that the superplasticity in a boron-doped Ni₃Al polycrystal results from a low flow stress and a high m value, which is caused mainly by DR.

Next, we will discuss superplasticity in Ni₃Al in comparison with other superplastic intermetallic compounds. Superplasticity in intermetallic compounds other than Ni₃Al has been reported in Fe₃(Al, Si)⁽⁷⁾, Ti₃Al⁽⁸⁾ and TiAl⁽⁹⁾. However, details on superplasticity in

Ti₃Al and TiAl have not been reported. Therefore, now let us consider superplasticity of Fe₃(Al, Si) in comparison with Ni₃Al.

The brittleness of Fe₃(Si_{0.65}Al_{0.35}), which is called "Sendust" and now used as a practical magnetic head material, has been surmounted by Hanada *et al.*^{(7)(38)–(42)} with controlling deformation conditions as well as melting and casting conditions. At an optimum condition of temperature and strain rate, Sendust can be deformed superplastically to elongation of about 100%.

Common features between Ni₃Al and Sendust are the considerably low flow stress and sufficient number of operating slip systems in the superplastic condition. In contrast, main differences between both alloys are an initial grain size (Ni₃Al: 1.6 μ m, Sendust: about 100 μ m) and dynamic restoration process during steady-state deformation (Ni₃Al: dynamic recrystallization, Sendust: both dynamic recovery and dynamic recrystallization). The occurrence of dynamic recovery in Sendust has been related to the change in dislocation appearance from a planar array of {110}<111> and {112}<111> slip systems depending on orientation at low temperatures to a non-planar array of them at elevated temperatures⁽³⁹⁾⁽⁴²⁾. Therefore, for Sendust, concentrated stresses arising from dislocation glide and GBS are easily accommodated by dynamic recovery within grains and dynamic recrystallization in the vicinity of grain boundaries. As a result, initial grain size is not an important controlling factor for superplastic deformation to occur. In contrast, for single crystals Ni₃(Al, Ti) with⁽⁴³⁾⁽⁴⁴⁾ and without boron^{(45)–(47)} a planar {111}<101> slip is operative even at high temperatures during deformation. Marked planarity of slip probably inhibits dynamic recovery process and causes stress concentrations at grain boundaries and eventually nucleates cracks in coarse-grained specimens⁽⁴⁸⁾. Therefore, an ultra fine grain size is the prime requirement for superplasticity in Ni₃Al-based compounds. From these view points, the overcoming of brittleness in intermetallic compounds seems to be directly related with the understanding of the deformation mechanism.

V. Conclusions

The results of this investigation on the condition and mechanism of superplasticity in a recrystallized Ni₃Al polycrystal doped with boron can be summarized as follows.

(1) Superplastic extension with a maximum elongation of 160% occurs under the condition of an ultra fine grain size (1.6 μ m), a low value of initial strain rate ($< 1.04 \times 10^{-4}$ s⁻¹) and a temperature of 973 K.

(2) From the TEM observation, dynamic recrystallization (DR) is found to be an important process operating during superplastic deformation. DR relieves a stress concentration arising from GBS and suppresses cavitation. Another important role of DR is to retard apparent grain growth.

(3) Further requirements for superplasticity are a low flow stress and a high m value which also contribute to reduce the cavitation.

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