

Readily superplastic forging at high strain rates in an aluminium-based alloy produced from nanocrystalline powders

K. TAKETANI, A. UOYA, K. OHTERA, T. UEHARA

Technical Research Laboratories, Yoshida Kogyo K.K., Kurobe, Toyama 938, Japan

K. HIGASHI

College of Engineering, Department of Mechanical Systems Engineering, University of Osaka Prefecture, Sakai, Osaka 593, Japan

A. INOUE, T. MASUMOTO

Institute of Materials Research, Tohoku University, Sendai 980, Japan

A high-strain-rate superplasticity at strain rates from 10^{-1} to 1 s^{-1} (which was many orders of magnitude higher than the strain rates in typical commercial superplastic alloys) was found in a new aluminium-based crystalline alloy, as-extruded Al–Ni–Mm crystalline and Al–Ni–Mm–Zr crystalline alloys (Mm = misch metal) fabricated by warm consolidation of their amorphous or nanocrystalline powders. The alloys were developed with very fine grained structures less than 100 nm in size, with a uniform distribution of both the Al_3Mm and the Al_3Ni particulates which were 70 nm in diameter. As a result of these specific microstructures, these alloys have superior mechanical properties at room temperature; for example the tensile strength is greater than 800 MPa and the Young's modulus is equal to 96 GPa. As-extruded workpieces of the alloys could be forged superplastically at a commercial production speed (less than 1 s) into complicated components with a con-rod shape, which exhibited good postdeformation mechanical properties.

1. Introduction

Recent advances in rapid solidification techniques have led to the development of new high-strength aluminium alloys [1]. Amorphous powders of Al–Ni–Ln (Ln = Y and misch metal) have been produced by a high-pressure-gas-atomization technique. As-extruded Al–Ni–Ln crystalline alloys, which were fabricated from amorphous powders, exhibited an excellent room-temperature strength [2], resulting from both grain refinement of the aluminium matrix and dispersion strengthening by intermetallic compounds. It was reported recently [3] that high strain rate superplasticity in an Al–14 wt % Ni–14 wt % Mm (Mm = misch metal) crystalline alloy was produced from amorphous powders. The maximum elongation of 540% was recorded at a high strain rate of $7 \times 10^{-1} \text{ s}^{-1}$ and 873 K. The optimum superplastic-strain-rate range in the Al–Ni–Mm crystalline alloy is many orders of magnitude faster than that for the best of the conventional commercial aluminium alloys. This result is due to the ultrafine grained structure of the Al–Ni–Mm crystalline alloy.

A new high-strength aluminium-based alloy, an Al–14.9 wt % Ni–3.3 wt % Mm–5.1 wt % Zr alloy, was also fabricated by warm consolidation from nanocrystalline powders [4]. Substituting Zr for Mm in Al–Ni–Mm alloy made the grain size finer, so that

it may be possible to develop superplastic Al–Ni–Mm–Zr alloys at a similar or a higher strain rate than that of the Al–Ni–Mm crystalline alloys. In this work, in order to confirm the presence of the high-strain-rate superplasticity in the as-extruded Al–Ni–Mm–Zr alloy, constant-true-strain-rate tests were carried out at a constant testing temperature of 873 K, which was the reported optimum superplastic temperature for the maximum elongation in the Al–Ni–Mm crystalline alloy [3]. Then it was demonstrated that the near shaped workpieces of the Al–Ni–Mm–Zr alloy fabricated by warm consolidation from nanocrystalline powders can be readily superplastically formed into a con-rod shape, and the postdeformation mechanical properties of the workpieces after forging were investigated.

2. Experimental procedure

The powders of Al–14.8 wt % Ni–6.6 wt % Mm–2.3 wt % Zr used in this work were produced by a high-pressure-gas-atomization technique at Yoshida Kogyo KK. As shown in Fig. 1, an electron micrograph taken from the electrolytically thinned powders showed that a typical nanocrystalline structure, about 10 nm in size, was observed in as-atomized Al–Ni–Mm–Zr powders. Black phases which are

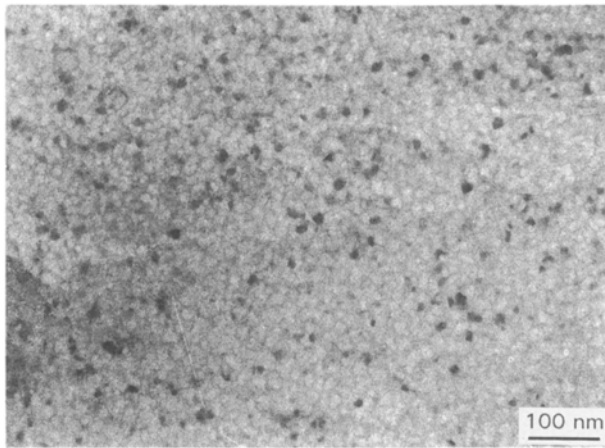


Figure 1 A typical nanocrystalline structure of the Al-14.8 wt % Ni-6.6 wt % Mm-2.3 wt % Zr alloy powders.

10 nm in size can also be observed in Fig. 1; they were metastable particles of Al_3Zr . The procedure used to prepare a bulky alloy was a powder-metallurgy method; Al-Ni-Mm-Zr nanocrystalline powders less than $45\ \mu\text{m}$ in size were sealed in a cylinder and degassed in vacuum, and then they were hot pressed. The compressed billets were extruded with a reduction ratio of 10:1.

Constant-true-strain-rate tests at a constant testing temperature of 873 K were carried out in air over a high strain rate range from 10^{-4} to $100\ \text{s}^{-1}$ in order to confirm the presence of high-strain-rate superplasticity in the Al-Ni-Mm-Zr alloy. The tensile specimens had a gauge length of 5 mm and a gauge diameter of 2.5 mm. The tensile axis was selected to be parallel to the extrusion direction.

The forging temperature condition had temperatures in the range 573–873 K, and the forging speed of 10 mm/s (corresponding to average strain rates of nearly $1\ \text{s}^{-1}$) was based on the results of tensile tests performed on the same material as the workpieces. A rod of the as-extruded Al-Ni-Mm-Zr alloy, which had a diameter of 13 mm, was heated very rapidly to the high forging temperature range given above, so it could be readily forged. After forging, the hardness, H_v , was measured by a Vickers hardness tester. Microstructures of the materials were observed by transmission electron microscopy (TEM) using the cross-sections of specimens.

3. Results and discussion

3.1. Microstructure

An as-extruded Al-Ni-Mm-Zr alloy had fine-grained structures less than 100 nm in diameter. Most of the grain boundaries were noted to have high-angle values, but grains with low-angle boundaries were also found occasionally. It has previously been reported [3] that a typical microstructure from an as-extruded Al-14 wt % Ni-14 wt % Mm crystalline alloy, produced from amorphous powders, consists of very fine recrystallized grains with a mean size of 100 nm and a uniform distribution of both Al_3Mm and Al_3Ni particulates (about 100 nm in size) and a high volume fraction of about 40%. As shown in Fig. 2, the structure of an as-extruded Al-Ni-Mm-Zr alloy produced

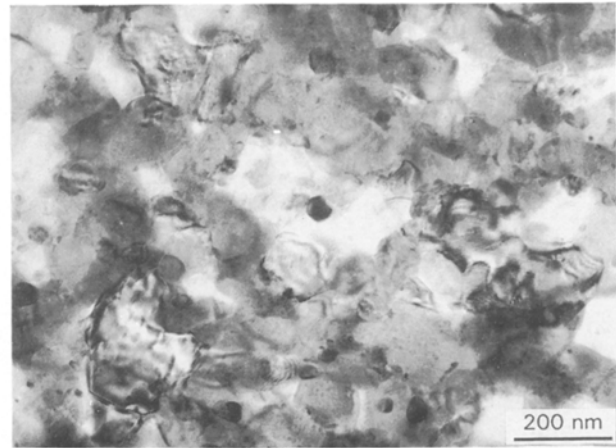


Figure 2 The microstructure of an as-extruded Al-14.8 wt % Ni-6.6 wt % Mm-2.3 wt % Zr alloy.

from nanocrystalline powders consists of finer grains than those of the Al-Ni-Mm crystalline alloy after extrusion. The mean value of the grain size was 50–80 nm in the as-extruded Al-Ni-Mm-Zr alloy. This fact shows that particles of Al_3Zr seem to be more efficient at preventing grain growth at high temperatures than those of intermetallics (including Ni or Mm). Intermetallic compounds of Al_3Mm and Al_3Ni , with a size of 10 ~ 50 nm, and precipitates of metastable Al_3Zr , less than 15 nm in size, were dispersed in the aluminium matrix with an almost homogeneous distribution. The volume fraction of total particles in the Al-Ni-Mm-Zr alloy is smaller (30%) than in the Al-Ni-Mm crystalline alloy. It is important to note that both a very fine-grained structure and an almost uniform distribution of the very fine particulates were obtained by extrusion only in both Al-Ni-Mm crystalline and Al-Ni-Mm-Zr alloys, without the need for any subsequent thermomechanical treatments.

Both the grain refinement of the aluminium matrix and the dispersion strengthening by intermetallic compounds are interpreted as contributing to the achievement of the high tensile strength in both the as-extruded Al-Ni-Mm crystalline and the Al-Ni-Mm-Zr alloys. Typical mechanical properties at room temperature reported previously [3, 4] are summarized in Table I for the as-extruded Al-Ni-Mm crystalline and for the Al-Ni-Mm-Zr alloys. However, these fine extruded structures were unstable at temperatures greater than about 773 K; the competition between grain growth and superplasticity that exists at high temperatures is addressed in ultrafine-grained Al-Ni-Mm crystalline and Al-Ni-Mm-Zr alloys. For example, as shown in Fig. 3, the fully annealed microstructure at 873 K for 1800 s for the Al-Ni-Mm-Zr alloy consists of grains with a mean size of $0.8\ \mu\text{m}$ and of particulates smaller than $0.5\ \mu\text{m}$ in size, and the high volume fraction of total particulates (about 30%).

3.2. High-temperature properties

Typical true-stress-true-strain curves for the Al-Ni-Mm-Zr alloy are shown in Fig. 4 at strain rates, $\dot{\epsilon}$, from 10^{-3} to $10\ \text{s}^{-1}$ at a test temperature of

TABLE I Typical mechanical properties at room temperature of the as-extruded Al-Ni-Mm and Al-Ni-Mm-Zr alloys

Alloy composition (wt%)	$\sigma_{0.2}$ (MPa)	σ_f (MPa)	ϵ (%)	E (GPa)	References
Al-14 Ni-14.6 Mm	815	909	0.7	104	[3]
Al-14 Ni-14 Mm	750	900	1.2	98	[2]
Al-14.9 Ni-3.3 Mm-5.1 Zr	886	1000	1.2	97	[3]
Al-14.8 Ni-6.6 Mm-2.3 Zr	823	892	4.9	96	[3]

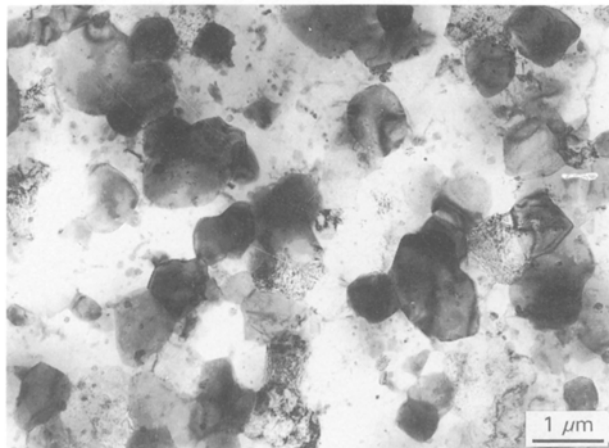


Figure 3 The microstructure of the Al-14.8 wt % Ni-6.6 wt % Mm-2.3 wt % Zr alloy fully annealed at 873 K for 1800 s.

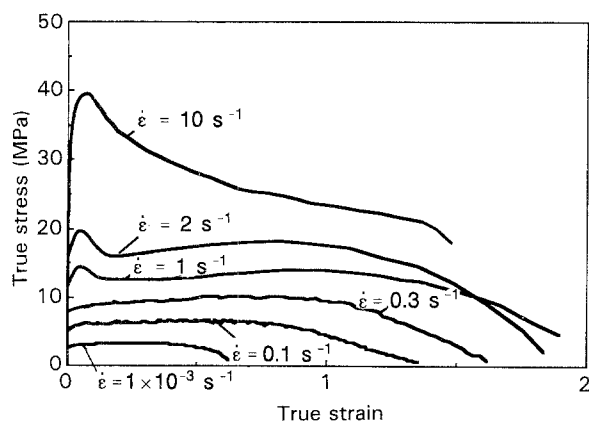


Figure 4 Typical true-stress-true-strain curves for the Al-14.8 wt % Ni-6.6 wt % Mm-2.3 wt % Zr alloy at strain rates from 10^{-3} to 10 s^{-1} at a testing temperature of 873 K.

873 K. In typical superplastic alloys such as I/M 7475 alloys [5], strain hardening was observed over the entire stress-strain curve during superplastic flow; this could be related to the grain growth which occurs continuously up to failure. For the Al-Ni-Mm-Zr alloy, curves for constant true strain rates below $1 \times 10^{-1} \text{ s}^{-1}$ exhibit an almost steady-state region followed by a region where gradual strain softening occurs prior to failure. In the strain range higher than 1 s^{-1} , strain softening occurs prior to failure. In the strain range higher than 1 s^{-1} , strain softening of the flow stress, suggesting that there is a possibility of microstructural evolution during deformation in samples deformed at strain rates over 1 s^{-1} . The shapes of true-stress-true-strain curves for the Al-Ni-Mm-Zr alloy are almost the same as those

previously reported [3] for the Al-Ni-Mm crystalline alloy.

The variation in the elongation and the flow stress for the Al-Ni-Mm-Zr alloy, as a function of the strain rate, is shown in Fig. 5a and b, respectively, for a testing temperature of 873 K; Fig. 5 also, includes the results for the Al-Ni-Mm crystalline alloy reported previously [3]. The flow stress for both the Al-Ni-Mm crystalline and the Al-Ni-Mm-Zr alloy increased with the strain rate in a typical sigmoidal curve, as has been previously observed for common superplastic materials. In the equation $\sigma = k\dot{\epsilon}^m$ (σ is the true stress, $\dot{\epsilon}$ is the true strain rate, and k is a constant incorporating the structure and temperature dependencies), the strain-rate sensitivity exponent, m , is the slope of this curve, $d(\ln \sigma)/d(\ln \dot{\epsilon})$. In general, large elongations are obtained in both temperatures and strain-rate ranges where high m -values are found.

For both Al-Ni-Mm crystalline and Al-Ni-Mm-Zr alloys deformed at a temperature of 873 K, a strain-rate sensitivity exponent of less than 0.2 was obtained in the low-strain-rate regime (below 10^{-2} s^{-1}). In the high-strain-rate range (from 10^{-1} to 100 s^{-1}), however, a high m -value of about 0.5 was

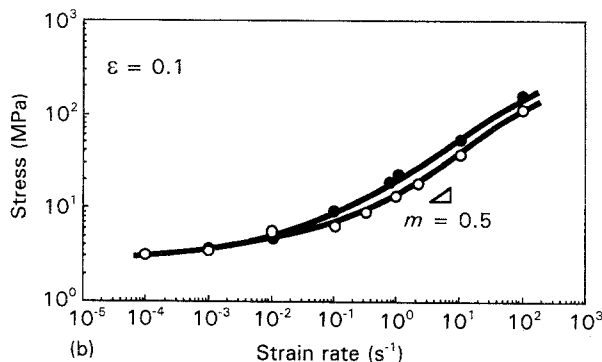
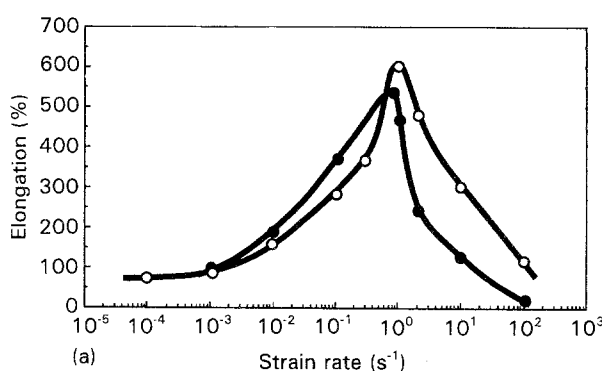


Figure 5 The variation in (a) the elongation (b) in the flow stress for (○) the Al-14.8 wt % Ni-6.6 wt % Mm-2.3 wt % Zr and (●) Al-14 wt % Ni-14 % Mm crystalline alloy as a function of the strain rate for a testing temperature of 873 K.

obtained. This high m -value of 0.5 suggests that grain-boundary sliding could be a primary deformation mechanism in both these alloys at high temperatures. In this region, the flow stress for the Al-Ni-Mm-Zr alloy is significantly lower than that for the Al-Ni-Mm crystalline alloys. This result is connected to the fact that the grain size of the Al-Ni-Mm-Zr alloy is finer than that of the Al-Ni-Mm crystalline alloy.

Elongations of less than 100% are obtained at strain rates below 10^{-3} s^{-1} in the Al-Ni-Mm-Zr alloy deformed at 873 K; however, the elongations increase with the strain rate, and values above 300% are found in the high-strain-rate range (over 10^{-1} s^{-1}). In particular, a maximum elongation of 600% is obtained at the very high strain rate of 1 s^{-1} . It is important to note that the optimum strain rate of 1 s^{-1} , for which the maximum elongation is obtained for the Al-Ni-Mm-Zr alloy, is slightly higher than that of $7 \times 10^{-1} \text{ s}^{-1}$ for the Al-Ni-Mm crystalline alloy. Elongation values for the Al-Ni-Mm-Zr alloy obtained at extremely high strain rates between 1 and 100 s^{-1} are larger than those for the Al-Ni-Mm crystalline alloy. It should be noted for both alloys that low elongations (more than 100%) were found at the highest strain rates, where the m -values are still high being nearly 0.5. This discrepancy may be associated with cavitation behaviour, which is directly related to the presence of the hard intermetallic compounds of Al_3Mm and Al_3Ni with a relatively large volume fraction. Since the applied stress increases for increasing strain rates, the increased local stress generated earlier at the hard-particle-matrix interfaces could lead to both this extensive cavitation and to the premature failure during superplastic flow. Cavitation work is in progress to verify this hypothesis. In any case, from the mechanical data from the constant-true-strain-rate tests, it was confirmed that high-strain-rate superplasticity was obtained in the as-extruded Al-Ni-Mm-Zr alloy produced from nanocrystalline powders at high strain rates of nearly 1 s^{-1} and at a high temperature of 873 K.

3.3. Superplastic-formation and post-deformation mechanical properties

An as-extruded Al-Ni-Mm-Zr alloy rod with a near shape was heated rapidly to high temperatures (between 573 and 873 K), then it was immediately superplastically forged into a con-rod shape, as shown in Fig. 6. A superplastic forming rate of an average strain rate of about 1 s^{-1} was determined on the basis of the results in Fig. 5; this corresponds to the optimum strain-rate range where large elongations were obtained in the Al-Ni-Mm-Zr alloy. The actual forging time was less than 1 s. The variation in the Vickers hardness of the forged workpieces is shown in Fig. 7 as a function of the forging temperature. The initial value of the Vickers hardness in the as-extruded workpieces was 240. However, the values of the Vickers hardness after forging tended to decrease with increasing forging temperatures. After forging at the reported optimum superplastic temperature of 873 K,

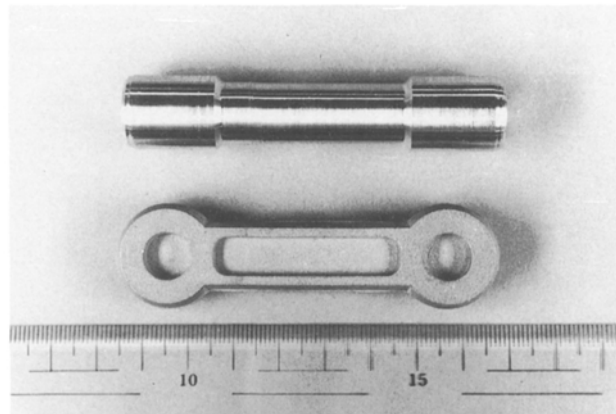


Figure 6 A rod with a near shape was superplastically forged into a workpiece with a con-rod shape and was heated rapidly to high temperatures then it was formed immediately.

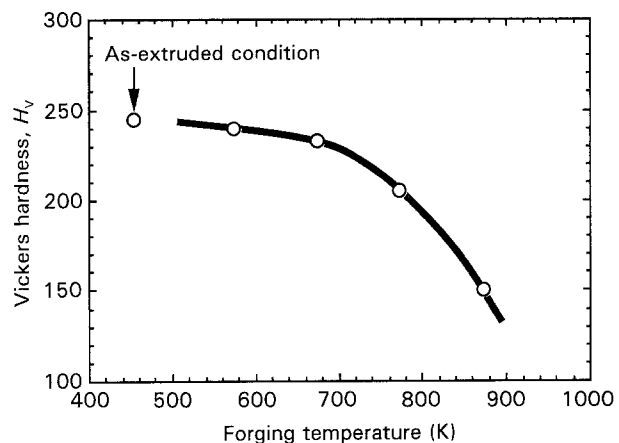


Figure 7 The variation in the Vickers hardness of the forged workpieces as a function of the forging temperature for the Al-14.8-Ni-6.6Mm-2.3Zr alloy.

the workpieces exhibited a low Vickers-hardness value of 150, resulting from the grain growth which occurs at high temperatures (as shown in Fig 3). Therefore, this kind of operation should lead a large reduction in postdeformation mechanical properties of the Al-Ni-Mm-Zr alloy. On the other hand, values of the Vickers hardness, greater than 235 – which are very close to the value of 240 which is obtained for the as-extruded condition – were obtained in the work-piece forged in the lower temperature range (below 673 K), which can be expected to achieve a similar degree in postdeformation mechanical properties.

The workpieces were successfully forged without any cracking in the temperature range 673–873 K, but they cracked near their surfaces when forged at 573 K. The lowest possible forging temperature in this investigation, 673 K, is much lower than the optimum superplastic deformation temperature of 873 K for both Al-Ni-Mm crystalline and Al-Ni-Mm-Zr alloys. No cracks were found inside the workpieces forged into a con-rod shape at 673 K (as can be seen in Fig. 8, which was obtained from a cross-section of the central region in the forged workpieces). Also it is clear from Fig. 7 that the edge section of the forged work-

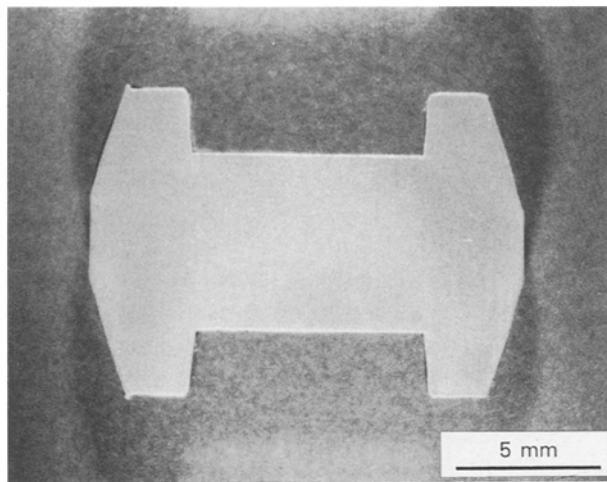


Figure 8 A cross-section of the central region in the forged workpieces.

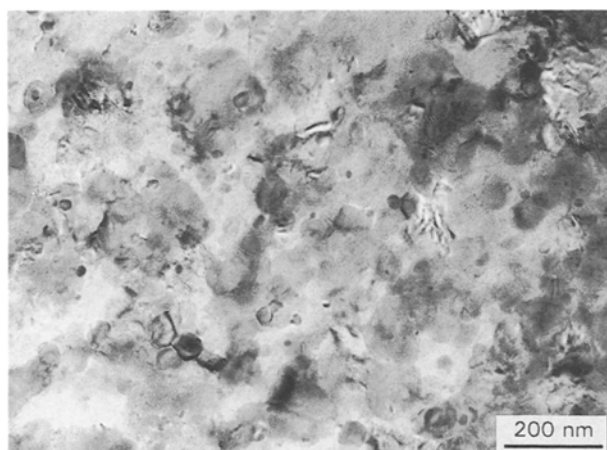


Figure 9 A typical microstructure from a workpiece forged superplastically at 673 K.

pieces was perfectly filled with the material. A typical microstructure from the workpieces was perfectly filled with the materials. A typical microstructure from the workpiece forged at 673 K remained almost equiaxed grains (as shown in Fig. 9), which suggests that the workpiece was forged superplastically at 673 K. Furthermore the grain size of the forged workpieces is nearly similar, or perhaps a little, coarser than that of the as-extruded workpiece, but the diameter of the particles clearly increased upon forging at 673 K. Typical values of the postdeformation mechanical properties at room temperature for the workpieces forged at 673 K are compared with values for the as-extruded condition in Table II. It should be noted that the mechanical properties of the as-extruded material used in the present work are inferior to the previous

TABLE II Post-deformation mechanical properties at room temperature of the as-forged Al-14.8 wt % Ni-6.6 wt % Mm-2.3 wt % Zr alloy

Alloy condition	$\sigma_{0.2}$ (MPa)	σ_F (MPa)	ϵ (%)	E (GPa)
As-forged at 673 K	729	784	2.0	96
As-extruded	781	828	1.9	96

data for the as-extruded Al-Ni-Mm-Zr alloy given in Table I. The tensile strength and the yield strength after forging at 673 K had lower values than the values for the as-extruded condition but remains a very large value at 780 or 730 MPa. The decrease in the post-deformation strength of the Al-Ni-Mm-Zr alloy forged at 673 K is considered to be due to microstructural changes in the growth of grains and/or dispersed particles.

4. Conclusions

A high-strain-rate superplasticity was observed for a Al-Ni-Mm-Zr alloy, and its maximum elongation of about 600% was obtained at 873 K and at a high strain rate of 1 s^{-1} ; this strain rate in the Al-Ni-Mm-Zr alloy was higher than the strain rate previously in Al-Ni-Mm crystalline alloy. The Al-Ni-Mm-Zr alloy was readily formed at high strain rates (the actual forging time was less than 1 s) and the mechanical properties of the formed component were almost identical to those of the as-extruded component. It is reasonable for hard forming materials, such as a Al-Ni-Mm crystalline or a Al-Ni-Mm-Zr alloy, to apply high-strain-rate superplastic formation to the production of components.

References

1. Y. TAKEDA, in Proceedings of 1993 Powder Metallurgy World Congress, edited by Y. Bando and K. Kosuge (Japanese Society of Powder and Powder Metallurgy, Kyoto, 1993) pp. 546-550.
2. K. OHTERA, A. INOUE and T. MASUMOTO, *Mater. Sci. Engng. A* **134** (1991) 1212.
3. K. HIGASHI, T. MUKAI, S. TANIMURA, A. INOUE, T. MASTUMOTO, K. KITA and J. NAGAHORA, *Scripta. Met. Mater.* **26** (1992) 191.
4. H. NAGAHAMA, K. OHTERA, K. HIGASHI, A. INOUE and T. MASUMOTO, *Phil. Mag. Lett.* **67** (1993) 225.
5. C. C. BAMPTON, A. K. GHOSH and M. W. MAHONEY, in "Superplasticity in aerospace-aluminum", edited by R. Pearce and L. Kelly (Ashford Press, Cranfield, 1985) pp. 1-35.

Received 16 September
and accepted 27 May 1994