Superplasticity of 20Cr–10Ni–0.7N (wt%) ultra-high nitrogen austenitic stainless steel

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Since nitrogen has been found to be an element which improves strength, nitrogen has been added to many austenitic heat-resistant stainless steels. However, this has a shortcoming in that ductility is lost. In the present study, ultra-high nitrogen stainless steel (UHNSS) of 20Cr–10Ni–0.7N (wt%) was prepared by a high-pressure melting method and the mechanical properties at elevated temperatures were investigated. High elongations were obtained on the occurrence of superplasticity of cold-rolled UHNSS at 1073 to 1273 K at low strain rates of the order of 10^{-4} sec^{-1}. The superplasticity was related to the precipitation of fine chromium nitrides which was promoted by cold rolling.

1. Introduction

Superplasticity of steels has been found in ultra-high carbon steel [1–3] and duplex stainless steels [4, 5]. In addition to the requirement of fine grains, the presence of fine spheroidized second-phase particles is required for fine structure superplasticity. Sherby and co-workers [1, 2] have reported that superplasticity appeared in ultra-high carbon steel from 1.3 to 1.9 wt% C. Precipitated cementite particles of the order of 0.1 to 0.5 μm are spheroidized uniformly during isothermal warm working at 923 K. The purpose of the cementite particles is to prevent grain growth of the fine matrix phase. Okade *et al.* [3] also revealed superplasticity in SUJ-2 steel (0.75C–0.29Si–0.32Mn–1.29Cr–Mo). In this material, cementite particles, precipitated after isothermal warm working, are spheroidized by the subsequent two-step heat treatments.

Superplasticity in 25Cr–6.5Ni–3Mo–0.14N (wt%) duplex stainless steel was reported by Maehara [4, 5]. The maximum elongation above 2500% is obtained under conditions where the σ-phase precipitation occurs by decomposition of δ-phase. The temperatures are high enough for dynamic recrystallization to take place. They emphasized the importance of prior treatment which controls the precipitation rate of σ-phase (the harder second-phase particles). Cold rolling done after the solution treatment of hot-rolled sheets promotes the precipitation of σ-phase. At the optimum strain rate for superplasticity, the local work hardening and the subsequent recrystallization of the softer matrix phase occurs around the σ-phase. The balancing of the two phenomena retains the fine austenitic grains and superplasticity occurs.

Nitrogen is very effective in austenite formation in steel, and it is a valuable element for use in austenitic stainless steels to improve the strength at elevated temperatures, but it usually lowers the elongation. However, if a large amount of nitrogen is included in austenitic stainless steel, fine nitrides will be precipitated and these will promote the superplasticity. Masumoto and Imai [6] explained a structure diagram of an 18Cr–Fe–Ni–N system alloy containing varying contents of nickel up to 20% and nitrogen up to 0.4%. They indicated that a dichromium nitride, Cr_2N, precipitates on grain boundaries on ageing below 1273 K in 18Cr–Fe–8Ni–0.29%N. Kikuchi *et al.* [7] investigated the ageing behaviour of 25Cr–20Ni–0.41N (wt%) stainless steels at temperatures in the range 973 to 1173 K. They observed that Cr_2N is precipitated by ageing in as-solution-treated specimens as a layer on grain boundaries or as a pearlite-like structure in the austenitic grains. However, the cold rolling of specimens after solution treatment produces finer precipitates which are dispersed uniformly in the grains.

In the present study, we investigated the superplastic behaviour in an ultra-high nitrogen stainless steel (UHNSS) which was prepared by a high-pressure melting method.

2. Experimental method

Samples were prepared as follows: Four ingots of 20Cr–10Ni–0.7N (wt%) stainless steel (UHNSS) of 10 kg weight were obtained at nitrogen pressures of 1 MPa. For comparison, an ingot of 20Cr–10Ni–low N was melted in air. The chemical compositions are listed in Table I. The ingots were hot rolled from 50 to 6 mm thickness after holding at 1523 K for 1 h. Then all the samples except two were subjected to solution treatment at 1573 K followed by cold rolling to 2 mm thickness. In order to examine the effect of the cold-rolling process, the other two samples were rolled in two steps: after solution treating at 1473 K, they were cold rolled to 3 mm, and then heat treated at 1273 K

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TABLE I Chemical compositions and heat treatments

No.*	Chemical compositions (wt %)	Reference
A86c	19.8Cr–9.9Ni–0.024N–0.014C–0.89Si–1.03Mn–0.010P–0.004S	As-cold-rolled
N47c	20.5Cr–10.1Ni–0.79N–0.011C–0.92Si–1.12Mn–0.007P–0.005S	
N47s		Solution treated
N54c	20.3Cr–9.98Ni–0.70N–0.013C–1.07Si–1.08Mn–0.007P–0.0043S	As-cold-rolled
N102c	20.5Cr–9.9Ni–0.68N–0.015C–1.7Si–1.17Mn–0.005P–0.0050S	
N102c2		Two-step cold-rolled
N108c2	20.6Cr–10.3Ni–0.63N–0.015C–1.02Si–1.01Mn–0.006P–0.0010S	

*A, melted in air; N, melted under high nitrogen pressure; c, as-cold rolled, 1573 K solution treatment (S.T.)–cold rolling (C.R.), 2ᵗ, s, solution treated, 1573 K S.T.–C.R., 2ᵗ-1573 K S.T.–water quench; c2, as-cold-rolled by two-step method, 1573 K S.T.–C.R., 3ᵗ-1273 K annealing–C.R., 2ᵗ.

followed by cold rolling to 2 mm thickness. Sample N47s was heat-treated at 1523 K after cold rolling to obtain the coarse recrystallized structure.

Superplasticity tests were done by tension. The specimens, as shown in Fig. 1, were machined from the cold-rolled sheets with the tensile axis coinciding with the rolling axis. An Instron-type machine was used for tensile tests. Cross-head speeds were changed from 0.45 to 45 mm min^{-1} and the temperature was varied from room temperature to 1473 K. The holding time at each deformation temperature was 2 min except in the case of specimen N47c in which it was 15 min. The strain-rate sensitivity exponent, m, in the equation $\sigma = K\dot{\varepsilon}^m$ was determined by change-in-strain-rate tests (Backofen method [8]) at 1273 and 1373 K, where σ is the flow stress, $\dot{\varepsilon}$ is the strain rate, and K is a material constant.

Microstructures of specimens were observed by optical microscopy and a scanning electron microscopy (SEM). The volume fractions of the precipitates were measured by the point count method.

3. Experimental results and discussion

Fig. 2 shows normal stress–strain curves of ultra-high and low nitrogen steels tested at various temperatures under a cross-head speed of 1 mm min^{-1} (initial strain rate $\dot{\varepsilon} = 8.33 \times 10^{-4}$ sec^{-1}). Above 1073 K, the elongations of the UHNSS (N102c2) became much higher than those of low nitrogen steel (A86c). An example of

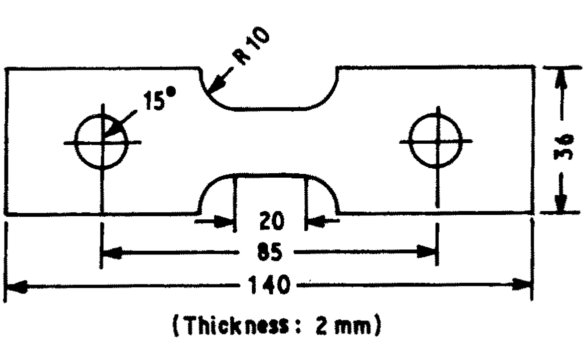

Figure 1 Shape of a specimen (mm in unit).

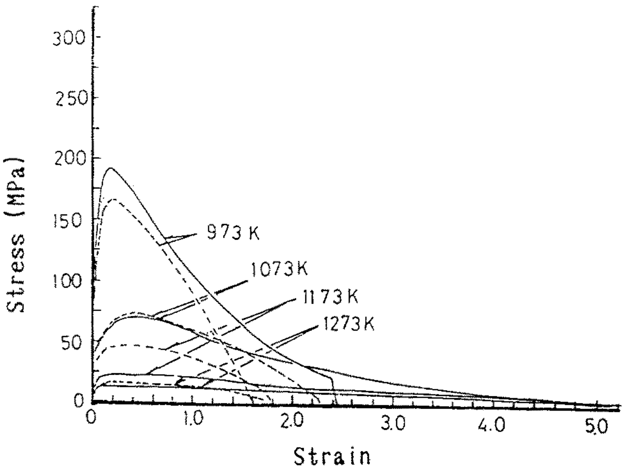

Figure 2 Example of normal stress strain curves. (——) UHNSS (N102c2), (- - -) low N steel (A28c). Cross-head speed = 1 mm min^{-1}.

a typical fractured sample obtained at 1073 K in the UHNSS is shown in Fig. 3 where elongation over 500% was attained.

Fig. 4 shows the influence of nitrogen content on elongation in the UHNSS. The elongations of low nitrogen steel (A86c) were nearly constant with a value of about 100% at temperatures higher than 973 K. On the contrary, those of high nitrogen steels became higher with increasing temperature and attained values of more than 200% at temperatures from 1073 to 1273 K.

Fig. 5 shows the influence of strain rate on elongation in UHNSS of sample N54c. At 45 mm min^{-1} cross-head speed ($\dot{\varepsilon} = 3.75 \times 10^{-2}$ sec^{-1}),

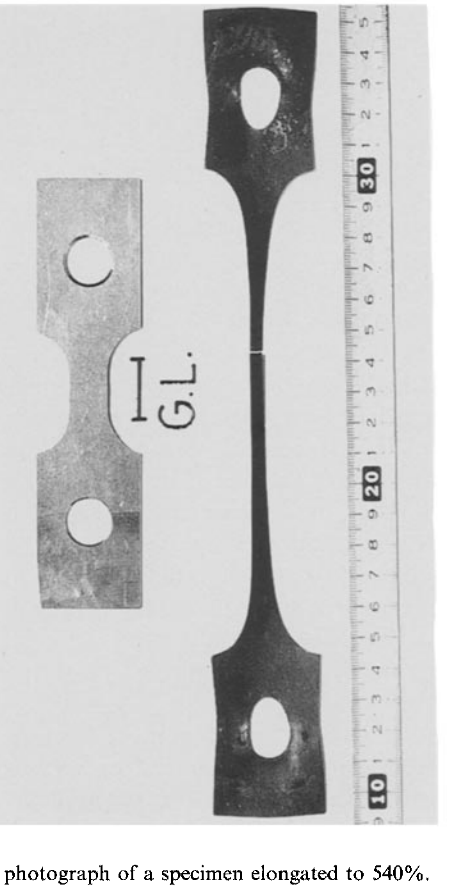

Figure 3 A photograph of a specimen elongated to 540%.

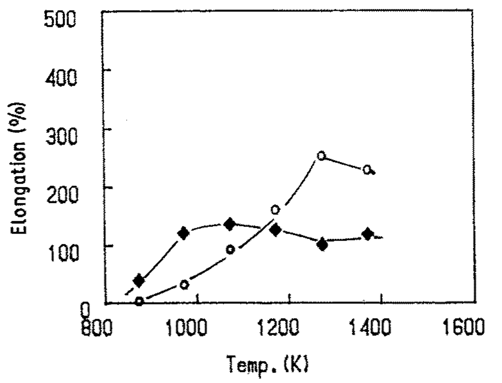

Figure 4 Influence of nitrogen content on elongation at elevated temperatures. Cross-head speed = 1.0 mm min^{-1}. (◆) A86c (0.024% N), (○) N102c (0.68% N).

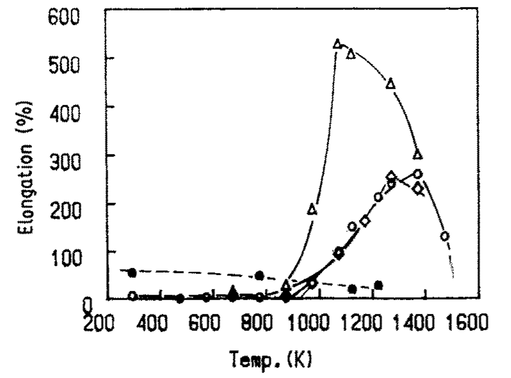

Figure 6 Influence of heat treatment and cold rolling on elongation at elevated temperatures. Sample number and cross-head speeds: (●) N47s, 4.5 mm min^{-1}; (◇) N102c, 1.0 mm min^{-1}; (○) N47c, 4.5 mm min^{-1}; (△) N102c2, 1.0 mm min^{-1}.

elongations were almost independent of temperature. These values were nearly equal to that of low nitrogen steel (A86c) in Fig. 4. As strain rate decreased, the elongation increased around 1273 K. The maximum value of elongation was 250% at 0.45 mm min^{-1} cross-head speed ($\dot{\varepsilon} = 3.75 \times 10^{-4}\,\text{sec}^{-1}$), indicating the occurrence of superplasticity.

Fig. 6 shows the influence of heat treatment and cold rolling on elongation in UHNSS. The elongation of the solution-treated sample (N47s) was very low and decreased slightly as temperatures rose. Those of as-cold-rolled specimens (N47c and N102c) were very low (a few per cent) below 873 K but they increased suddenly above 973 K and slightly decreased over 1373 K. The maximum elongation of as-cold-rolled materials was about 250%. This tendency appeared much stronger in the two-step cold-rolled specimen (N102c2), but the temperature at maximum elongation decreased by 200 K compared with as-cold-rolled specimens. The value amounted to 527% at 1073 K.

Fig. 7 shows the strain-rate sensitivity exponents, m, measured by the Backofen method for two-step cold-rolled UHNSS (N108c2) plotted against strain rate as a function of temperature. These m-values exceeded 0.35 in the strain rates below $2 \times 10^{-3}\,\text{sec}^{-1}$ at 1373 K and below 1×10^{-2} at 1273 K. The maximum value of 0.64 was obtained at 1273 K, in $\dot{\varepsilon} = 7 \times 10^{-4}\,\text{sec}^{-1}$.

Fig. 8 shows scanning electron micrographs of two-step cold-rolled UHNSS (N102c2); Fig. 8a shows the polished surface before deformation and Fig. 8b the fractured surface deformed at 1273 K (elongated to 450%). A large amount of fine precipitates (of the order of 0.2 to 1 μm) are seen in Fig. 8a. The grain sizes of the matrix are about 3 μm in the former and 3 to 10 μm in the latter. In reference to the phase diagram of 18Cr–Fe–8Ni–N constructed by Masumoto and Imai [6], it is most probable that the precipitates are Cr_2N, although we could not identify the structure. The volume fractions of precipitates were about 24% and it was nearly equal before and after deformation. This reveals that most of the chromium nitrides had precipitated during annealing before the second cold rolling, as is the case in UHNSS of 25Cr–20Ni–0.41N (wt %) examined by Kikuchi *et al.* [7]. In Fig. 6, the maximum elongation of the two-step cold-rolled sample (102c2) became higher and the temperature at the peak shifted to the lower temperature side compared with as-cold-rolled samples. The features indicate that the two-step cold-rolled samples had finer grain size than as-cold-rolled samples. It follows from these facts that the second cold rolling promoted the refining of grain size by the precipitation of fine nitrides.

4. Conclusion

It has become clear that a cold-rolled ultra-high nitrogen stainless steel of 20Cr–10Ni–0.7N (wt %) exhibits superplastic behaviour. This was particularly enhanced by two-step cold rolling. The maximum elongation

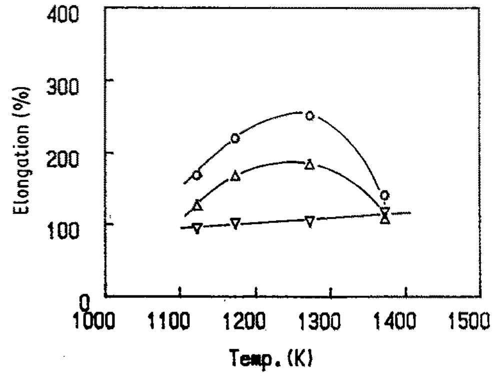

Figure 5 Influence of strain rate on elongation for N54c (0.70% N). Cross-head speeds (initial strain rates): (▽) 45 mm min^{-1} ($3.75 \times 10^{-2}\,\text{sec}^{-1}$); (△) 4.5 mm min^{-1} ($3.75 \times 10^{-3}\,\text{sec}^{-1}$); (○) 0.45 mm min^{-1} ($3.75 \times 10^{-4}\,\text{sec}^{-1}$).

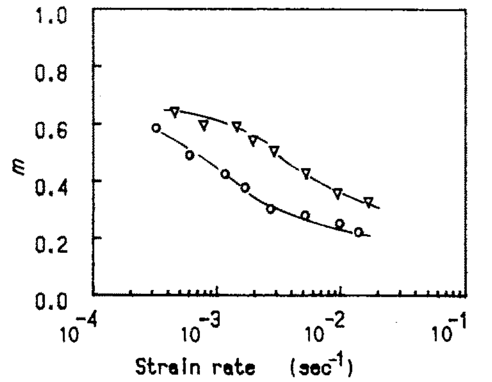

Figure 7 Strain-rate sensitivity exponent (m-values) measured by the Backofen method, (▽) at 1273 K, (○) at 1373 K.

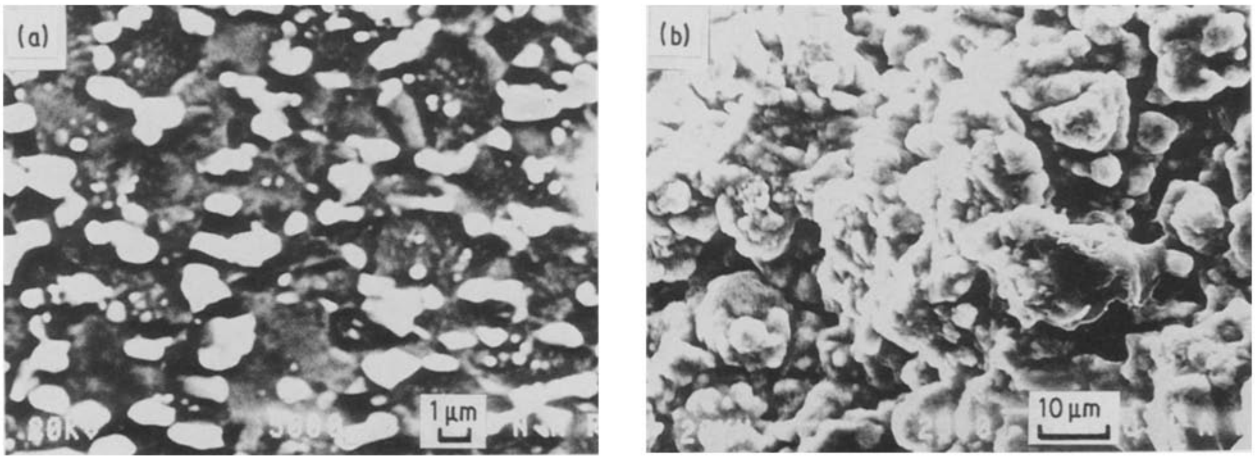

Figure 8 Scanning electron micrographs of UHNSS (N102c2): (a) Polished surface before deformation at 1073 K; (b) fractured surface deformed at 1273 K (elongated to 450%).

was obtained at 1073 K and at low initial strain rate $\dot{\varepsilon} = 7 \times 10^{-4}\,\mathrm{sec}^{-1}$ in two-step cold-rolled steels.

The superplasticity of this steel was associated with the precipitation of fine chromium nitrides which was promoted by the cold rolling.

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References
1. O. D. SHERBY, B. WALSER, C. M. YOUNG and E. M. CADY, *Scripta Metall.* **9** (1975) 569.
2. B. WALSER and O. D. SHERBY, *Met. Trans. A* **10A** (1979) 1461.
3. M. OKADE, M. TOKIZANE and O. D. SHERBY, *Tetsu-To-Hagane* **67** (1981) 141.
4. Y. MAEHARA, *ibid.* **70** (1984) 2168.
5. Y. MAEHARA and Y. OHMORI, *Sumitomo Search* **31** (1985) 147.
6. T. MASUMOTO and Y. IMAI, *Nippon Kinzoku Gakkaishi* **33** (1969) 1364.
7. M. KIKUCHI, T. SEKITA, S. WAKITA and R. TANAKA, *Tetsu-To-Hagane* **62** (1976) 981.
8. W. A. BACKOFEN, I. R. TURNER and D. H. AVERY, *Trans. ASM* **57** (1964) 980.

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