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Modification of anisotropic mechanical properties in recrystallized oxide dispersion strengthened ferritic alloy

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Abstract

Anisotropic mechanical properties of recrystallized commercial grade Fe–20%Cr ferritic oxide dispersion strengthened alloys were investigated. Recrystallized microstructures were modified through preannealing and partial-recrystallization heat treatments, resulting in more isotropic mechanical properties in both longitudinal and transverse directions.

Keywords: Recrystallization kinetics; Iron alloys; Oxide dispersion strengthened; Texture; Low temperature deformation

1. Introduction

Oxide dispersion strengthened (ODS) ferritic/martensitic steels have been investigated for high temperature applications [1–3]. In recent times, their better swelling resistance as well as their superior creep strength at elevated temperatures than austenitic steels have made them the prospective cladding materials for the next generation of nuclear power plants [4–6]. The development of ODS ferritic/martensitic steels has been mainly attempted in the field of fast reactor fuel cladding and fusion reactor materials applications. Thin-walled cladding tubes have been manufactured by mechanical alloying, hot extrusion and warm rolling processes. These materials had a strong anisotropy of mechanical properties due to the highly elongated grain structure along the rolling direction limiting the application of thin-walled cladding tube [4]. The effects of deformation, recrystallization, and phase transformation processes on the recrystallized grain morphology of ODS alloys have been reported [7]. The preannealing was effective in refining the recrystallized microstructure of MA956 alloy even though the fine grains did not tend to be equiaxed. In the case of MA957, equiaxed grains were obtained by the preannealing due to it having a lower oxide content than MA956. Bhadeshia [8] provided reviews on the unusual recrystallization behaviour of Fe-based MA–ODS alloys and reported that a preannealing did not affect the recrystallized grain structure in PM2000 alloy which has been considered as a highly recommended Fe-based MA–ODS alloy for reactor cladding materials [9,10].

In this study, the anisotropy and temperature dependence of tensile properties of extruded and recrystallized Fe–20%Cr ferritic ODS alloys have been characterized. Then, the effects of partial-recrystallization heat treatments, which were thought to be more effective than preannealing for the control of recrystallized microstructure, were investigated and the improvement of anisotropic mechanical properties via the heat treatments was discussed.

2. Experimental procedures

Intermediate products of a commercial Fe–20%Cr ODS alloy, PM2000™, were used in this study. This material is

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an oxidation resistant and creep resistant ferritic Fe–Cr–Al based alloy and its nominal composition (wt.%) is Fe–20Cr–5.5Al–0.5Ti–0.5Y_2O_3. The diameter of initial as-forged bar after mechanical alloying was 380 mm. Extruded bars with various reductions of the area of 81.8%, 93.1%, 97.5% and 99.9% were provided by Plansee GmbH. Texture analyses were carried out by the X-ray pole figure. {110}, {200} and {211} pole figures were obtained by Schultz reflection method on the sample surface normal to the extrusion axis with X-ray diffraction (Rigaku D/max-IIIC, Cu K_α).

Samples were cut from the surface of the as-received bar specimens. Recrystallization temperatures of the extruded bars were determined from the recrystallization grain fronts of the bars positioned in a tube furnace with a fixed thermal gradient. Microstructures of the extruded and the recrystallized bars were observed by optical microscopy and transmission electron microscopy (TEM). Extruded bars were preannealed at 1100 °C at which the recovery was presumably completed in 200 h and the partial-recrystallization heat treatment was carried out at 1350 °C for 5, 10, 20, 30, 45 or 60 min. The fraction of the recrystallized grain was measured using an image analysis system. Tensile tests in both longitudinal and transverse directions were conducted with the strain rate of $8 \times 10^{-4}\ s^{-1}$ after holding the specimens at the test temperature of 25–800 °C for 20 min. Tensile data of three samples in each condition were averaged.

3. Results and discussion

3.1. Anisotropy and temperature dependence of tensile properties of Fe–20%Cr ODS alloys

The typical microstructure of the extruded ferritic ODS alloy consisted of lightly elongated grains of 1–3 μm in diameters and finely dispersed Y_2O_3 particles of about 20 nm as observed by TEM. The tensile behaviour of extruded alloy as shown in Fig. 1 is similar in the longitudinal and transverse directions owing to the small and more or less equiaxed grains. Extruded specimens have a strong {111} γ fiber texture that is typical in deformed Fe–Cr steels. Strain hardening anisotropy in extruded conditions can be affected by texture [11], but the texture-dependent plastic anisotropy is outside the scope of our work and is not treated in more detail. Extruded bars were recrystallized to coarsen the grains and to reduce the amount of grain surface per unit volume by about 2–3 orders of magnitude for high temperature applications. In fully recrystallized conditions, they showed strong anisotropic mechanical properties as shown in Fig. 2. Yield strength and elongation in the transverse direction is lower than in the longitudinal direction both at room temperature and 600 °C. Tensile tested microstructure in transverse direction showed a sharp flat fracture surface in a similar fashion to a brittle fracture mode (Fig. 3). The strong anisotropy of mechanical properties in the fully recrystallized condition is related to the large elongated grain structure along the extrusion direction. Also, when the longitudinal yield strengths of the extruded and the recrystallized specimens were compared (Fig. 4), the yield strength below 700 °C showed a large difference between the extruded specimens and the recrystallized ones. However, the yield strength gap was significantly reduced above 700 °C and that is attributed to the reduced grain boundary strengthening effect by the accelerated diffusion through the grain boundaries at high temperature. This was indirectly confirmed by the larger void formation on the fracture surface with the higher test temperature.

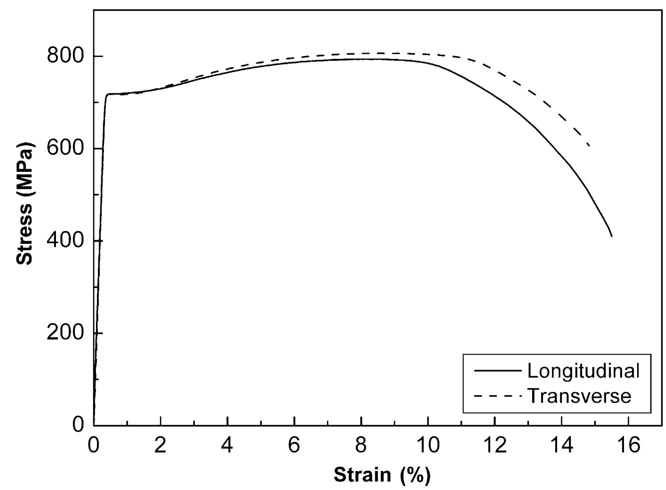

Fig. 1. Stress–strain curves of 97.5% extruded Fe–20%Cr alloy in longitudinal and transverse directions (test temperature: 25 °C).

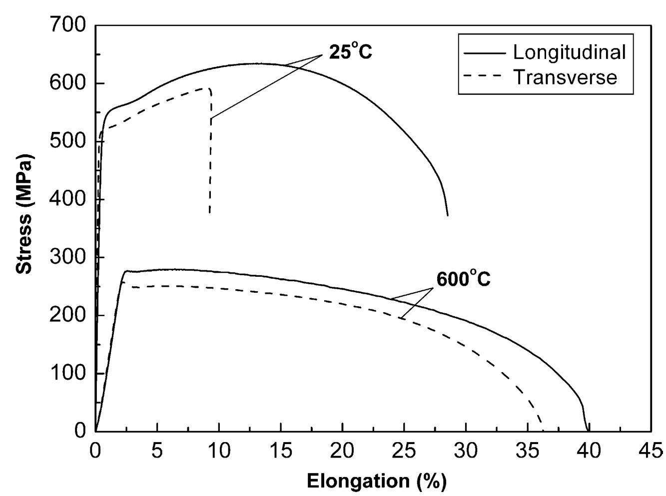

Fig. 2. Stress–strain curves in recrystallized condition of 97.5% extruded Fe–20%Cr alloy in longitudinal and transverse directions (test temperatures: 25, 600 °C).

3.2. Effect of partial-recrystallization after preannealing on mechanical properties of Fe–Cr MA–ODS alloy

As shown in Fig. 3, a typical recrystallized microstructure of Fe–20%Cr ODS alloy with high Y_2O_3 content has highly elongated and very large grains due to the longitudi-

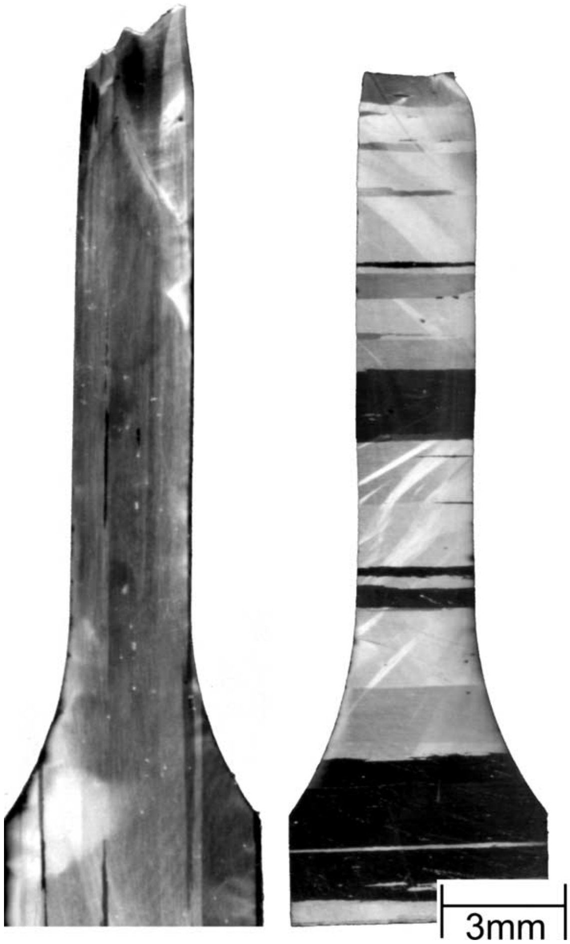

Fig. 3. Cross-sectional macrostructure of tensile tested specimens (Fig. 2) in ambient temperature.

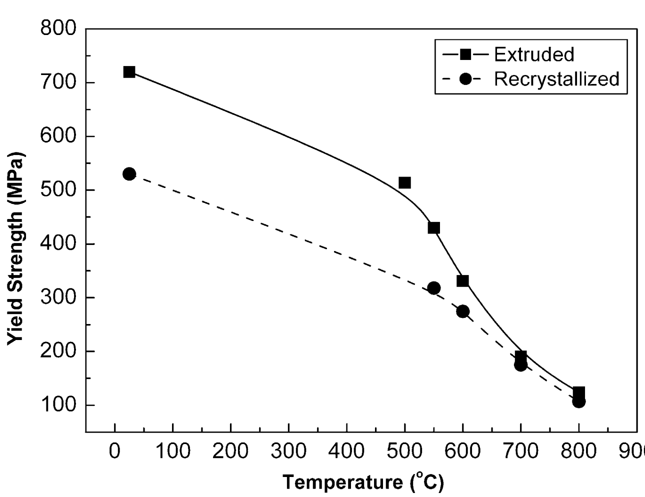

Fig. 4. Temperature dependence of yield strength of Fe–20%Cr alloys in extruded and recrystallized conditions.

nally aligned dispersoids that make the grain shape difficult to be adjusted into the equiaxed ones [12,13]. Hence, the mixed microstructures of the deformed grains and the recrystallized grains, formed by the partial-recrystallization after preannealing, would be preferable to modify the anisotropic microstructure as well as to enhance the mechanical properties at an intermediate temperature range (400–600 °C).

When the extruded bars were isothermally heat-treated at 1100 °C, the recovery level estimated by the hardness measurement was saturated after 200 h. Recrystallization temperatures of the extruded bars were decreased by the preannealing treatment as shown in Fig. 5. These observations are consistent with the behaviour of other preannealed Fe-based MA–ODS alloys [3,7]. The stored energies of Fe–20%Cr ODS alloys decrease during preannealing, whereas the recrystallization becomes easier. It is clear that the preannealing leads to a significant change in the recrystallization microstructure. Several recovery mechanisms including a subgrain coalescence would be activated during preannealing and it is seen that factors other than stored energy alone determine the recrystallization kinetics. Some previous results reported that the strengthening of {1 1 1} texture, which is typically evolved in deformed Fe–Cr MA–ODS alloy, make the nucleation easier by preannealing [7,14]. Fig. 6 shows the air-quenched microstructure after heat treatment for various times (0–45 min) at 1350 °C following the preannealing treatment. Recrystallization volume fraction of 50% ($X_1 \approx 0.5$) was obtained after the partial-annealing at 1350 °C for 20 min, and the recrystallization was completed after 30 min. Although a prolonged preannealing treatment itself could develop the mixed microstructure containing recrystallized grains in extruded grains [3], the partial-recrystallization treatment was found to be more effective to obtain the mixed microstructure.

Yield strengths of various specimens at 600 °C are compared in Fig. 7. The average yield strength of a fully recrystallized specimen shows differences of about 20 MPa

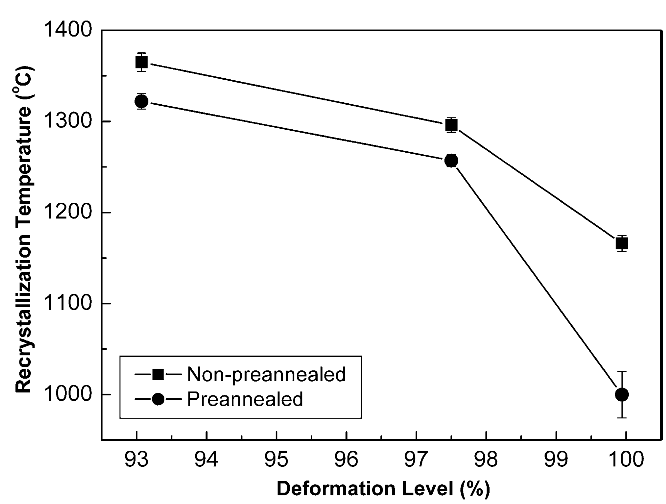

Fig. 5. Preannealing effect on the recrystallization temperature in Fe–20%Cr alloys with various extrusion ratios. Recrystallization temperatures were determined from the locations of 10 mm depth of as-extruded bar surface, except for the 99.9% extruded one obtained from the center of the recrystallization front.

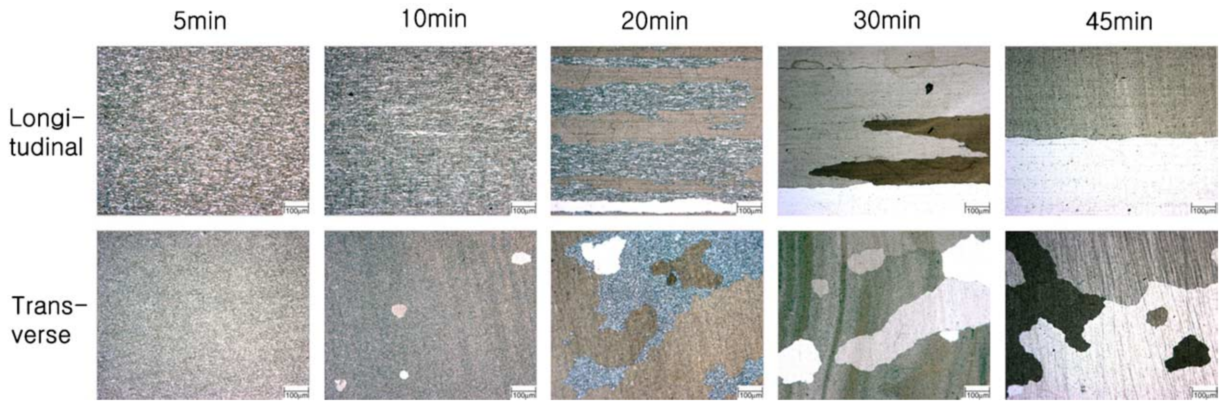

Fig. 6. Optical micrograph of air-quenched specimens after heat treatment for various times (0–45 min) at 1350 °C following preannealing at 1100 °C for 97.5% extruded specimen.

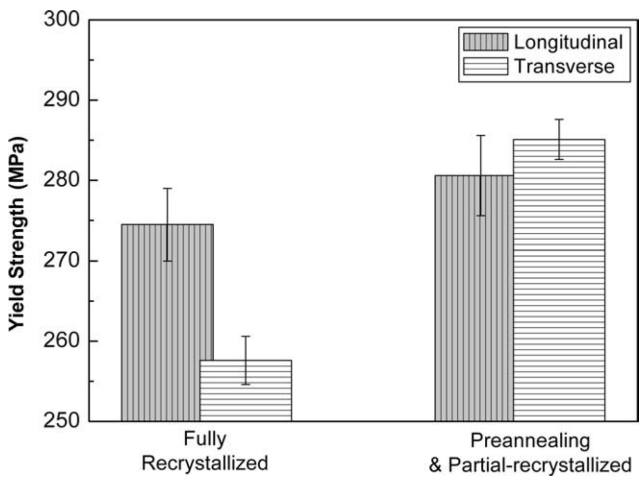

Fig. 7. Yield strengths of fully recrystallized specimen for 60 min at 1350 °C and partially recrystallized one for 20 min at 1350 °C following 1100 °C preannealing for 97.5% extruded bar (test temperatures: 25, 600 °C).

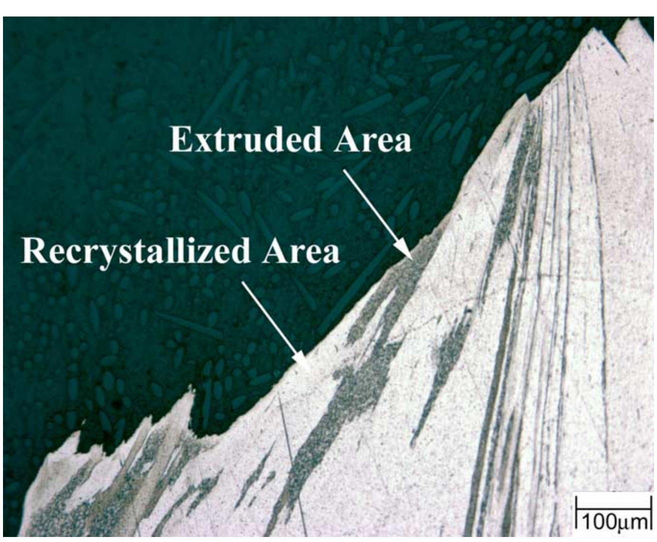

Fig. 8. Longitudinal cross-sectional microstructure of 600 °C tensile tested specimens in preannealed and partial-recrystallized specimen for 97.5% extruded Fe–20%Cr alloy.

depending on the loading direction. However, the partially recrystallized specimens following the preannealing treatment show much smaller differences (about 5 MPa) between both directions. Fig. 8 shows a cross-sectional microstructure of the fracture surface of a partially recrystallized specimen. The combination of the preannealing and the partial-recrystallization treatment developed an enhanced nucleation and a limited growth so that the enhanced isotropic mechanical properties were accomplished through the mixed microstructure of extruded and recrystallized area.

4. Conclusions

Microstructures and mechanical properties of Fe–20%Cr ferritic ODS alloys have been investigated in extruded and recrystallized conditions. While the extruded samples showed relatively isotropic mechanical properties, the fully recrystallized ones showed strong anisotropic mechanical properties. The yield strength and elongation in the transverse direction were lower than those in the longitudinal direction, both at room temperature and 600 °C. Additionally, as the temperature dependences of yield strength in extruded and recrystallized conditions were compared, the yield strength of the recrystallized specimen below 700 °C was lower than that of the extruded condition. Therefore, preannealing followed by partial-annealing was applied in order to improve the anisotropy and loss of mechanical properties in the fully recrystallized condition. Preannealing reduced the recrystallization temperature in all extruded bars. A mixed microstructure consisting of deformed and recrystallized grain was obtained by partial-recrystallization heat treatment at 1350 °C for 20 min. As a result, enhanced isotropic mechanical properties were accomplished in both longitudinal and transverse directions by the combined effects of preannealing and partial recrystallization.

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