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ANALYSIS OF STRENGTH AND DUCTILITY OF PARTIALLY RECRYSTALLIZED Cu-4.5 wt.% Al

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ABSTRACT

A series of partially recrystallized Cu-4.5 wt.% Al samples with different volume fractions of recrystallized grains were prepared by dynamic plastic deformation at room temperature followed by annealing treatments. The samples consist of nanotwins and micrometer recrystallized grains. Tensile tests showed that the flow stress decreases approximately linearly as a function of the volume fraction of recrystallization, whereas the uniform elongation does not increase significantly until the volume fraction exceeds a certain value. The results were analyzed in comparison with the tensile behavior of an idealized model composite whose stress-strain curve was deduced from that of its two constituting components based on the rule of mixtures.

1. INTRODUCTION

While nanostructured and ultrafine-grained metals are extremely strong, they usually exhibit a very limited ductility which hinders their practical applications (Koch, Morris, Lu and Inoue 1999). Accordingly, many efforts have been made to improve the ductility of nanostructured and ultrafine-grained materials, but results have not been conclusive (Huang, Hansen and Tsuji 2006; Tsuji, Ito, Saito and Minamino 2002; Wang, Chen, Zhou and Ma 2002; Zhao, Topping, Bingert, Thornton, Dangelewicz, Li, Liu, Zhu, Zhou and Lavernia 2008). In a partially recrystallized structure developed by annealing of ultrafine-grained Cu samples, a promising strength-ductility combination (Wang, Chen, Zhou and Ma 2002) was obtained, where a significant increase in tensile uniform elongation (ε_u) was obtained when the recrystallized volume fraction (f_{RX}) exceeded 25%. However, in partially recrystallized Cu processed by dynamic plastic deformation (DPD) or quasi-static compression with subsequent annealing, no obvious improvement of ε_u was observed until f_{RX} exceeded 80% (Li, Zhang, Tao and Lu 2008). In partially recrystallized 316L stainless steel samples and Fe-25Mn steel samples subjected to DPD and subsequent annealing, ε_u starts to increase at small f_{RX} (Wang, Tao and Lu 2012; Yan, Liu, Tao and Lu 2012).

In the present work, a partially recrystallized microstructure in Cu-4.5 wt.% Al samples is prepared by DPD and subsequent annealing, and its mechanical properties are measured and analyzed in terms of the strength and ε_u as a function of f_{RX}.

2. EXPERIMENTAL

The material used in the present investigation was a single phase Cu-4.5 wt.% Al alloy with a stacking fault energy reported to be about 12 mJ/m^2 (Murr 1975). Prior to the DPD treatment, the alloy was annealed at 1123 K for 2 h, resulting in a coarse-grained structure with an average grain size of about 200 μm. Cylinders 9 mm in diameter and 12 mm in height were subjected to the DPD treatment on a dynamic compression facility; the cylindrical specimen on a lower anvil was impacted by an upper anvil at a high loading rate at room temperature (RT). The plastic strain was controlled to be 0.2~0.3 at each impact with a strain rate in a range of 10^2-10^3 s^{-1}. The strain is defined as $\varepsilon = ln\ (L_0/L_f)$, where L_0 and L_f are the heights of the cylinder before and after the impact, respectively. The samples in the present work were deformed to a high accumulative strain of $\varepsilon = 1.7$.

Isochronal annealing of the DPD samples were performed in silicone oil baths at various temperatures ranging from 250 to 310 ℃ for 20 minutes. The samples were cooled in the air after annealing.

The microstructures were characterized by scanning electron microscopy (SEM) with electron channeling contrast (ECC) imaging in a LEO SUPRA 35 microscope and by transmission electron microscopy (TEM) on a JEOL 2010 microscope. Specimens were examined along the longitudinal section containing the compression axis.

Mechanical properties were evaluated by uniaxial tensile tests on an Instron 5848 MicroTester (2 kN) at a strain rate of 10^{-3} s^{-1} at ambient temperature. Dog-bone shaped tensile specimens were prepared along the radial direction of the pancake-like samples, with a gauge length of 5 mm and a cross-sectional area of 1×1 mm^2. A contactless MTS LX300 laser extensometer was used to calibrate and measure the tensile strain.

3. RESULTS

The general microstructural features of the Cu-4.5 wt.% Al samples processed by DPD to =1.7 are presented in Fig. 1a, which shows that rhomboidal prisms containing a high density of deformation twins are embedded in nano-sized grains. As shown in Fig. 1b, fine parallel traces are visible in many of the prism-like regions, indicating the existence of twin/matrix (T/M) lamellae. The volume fraction of the regions with a high density of deformation twins is about 18% as measured from SEM-ECC images. These microstructural features were also observed by TEM observations. A high density of dislocations was observed in the T/M lamellae. The thicknesses of the T/M lamellae were measured using TEM images and a thickness distribution was made based on volume fraction. The distribution was well fitted using a lognormal function with mode = 6.4 nm, mean = 26.1 nm, and standard deviation = 32.5 nm. The nano-sized grains were measured using TEM images to have an average transverse size of 50 nm.

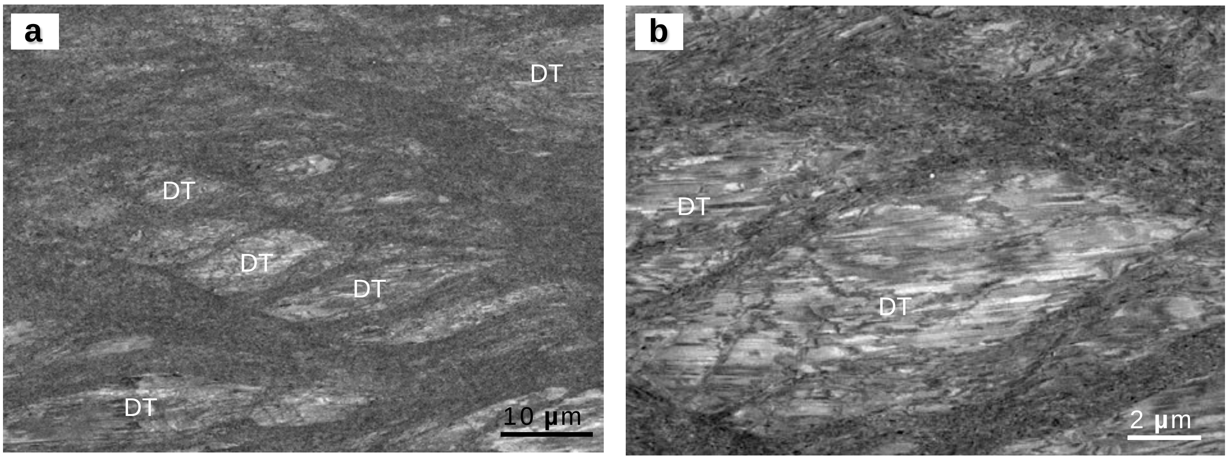

Fig. 1. SEM-ECC observations of Cu-4.5 wt.% Al samples processed by DPD to ε=1.7. (a) The overall microstructure; (b) a closer observation. "DT" indicates regions with high density of deformation twins.

Annealing of the above DPD samples at various temperatures ranging between 250 and 310 ^{o}C for 20 minutes leads to partial recrystallization of the samples to different extents. The very early stage of recrystallization was observed in the samples annealed at 250 ^{o}C. Figure 2a shows a typical SEM-ECC observation of the samples. The occurrence of isolated recrystallized grains is evident, as indicated by the dashed ellipses. The volume fraction of the recrystallized grains was measured to be f_{RX}=1%. Annealing twins are observed in most of the recrystallized grains, as is characteristic of materials with a low stacking fault energy. Nucleation of these recrystallized grains is observed inside the regions of nano-sized grains which can be either distant or adjacent to the highly twinned regions, but is not found inside the highly twinned regions. A closer SEM-ECC observation of the microstructure is given in Fig. 2b. The recrystallized grains, marked by the dashed ellipses, are formed in the nano-sized grains adjacent to the highly twinned regions.

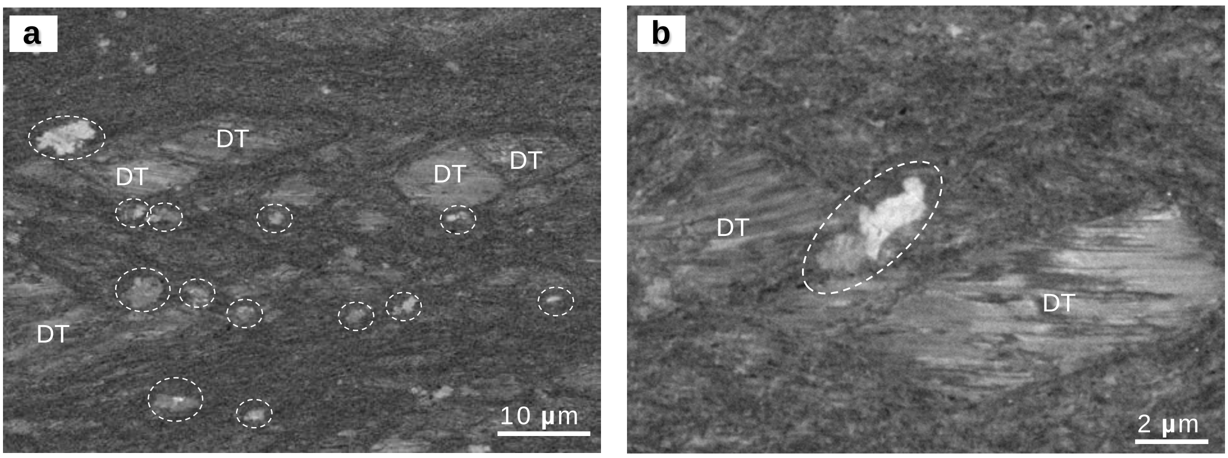

Fig. 2. SEM-ECC observations of Cu-4.5 wt.% Al samples processed by DPD to ε=1.7 and subsequently annealed at 250 ^{o}C for 20 minutes. (a) The overall microstructure; (b) a closer observation. "DT" indicates regions with high density of deformation twins. The dashed ellipses mark recrystallized grains.

As the annealing temperature increases, the volume fraction of the recrystallized grains increases. The recrystallization nucleation and growth occurred mainly within the region of nano-sized grains. A more advanced stage of recrystallization is observed in the samples annealed at 300 ^{o}C. As shown by the SEM-ECC image in Fig. 3a and b, the nano-sized grains in the as-deformed state have been consumed by recrystallized grains, whereas many highly twinned regions are retained. Some of the recrystallized grains are found to have grown into the highly twinned regions, but recrystallized grains inside the highly twinned regions are scarcely observed. The recrystallized grains have a volume fraction of 85.7% in this sample. The samples were also characterized by TEM. The high density of dislocations observed in the as-deformed samples is retained in the T/M lamellae. The thickness distribution of the T/M lamellar is well fitted by a lognormal function with mode = 7.3 nm, mean = 23.1 nm, and standard deviation = 24.9 nm, and is therefore little changed compared with the as-deformed samples.

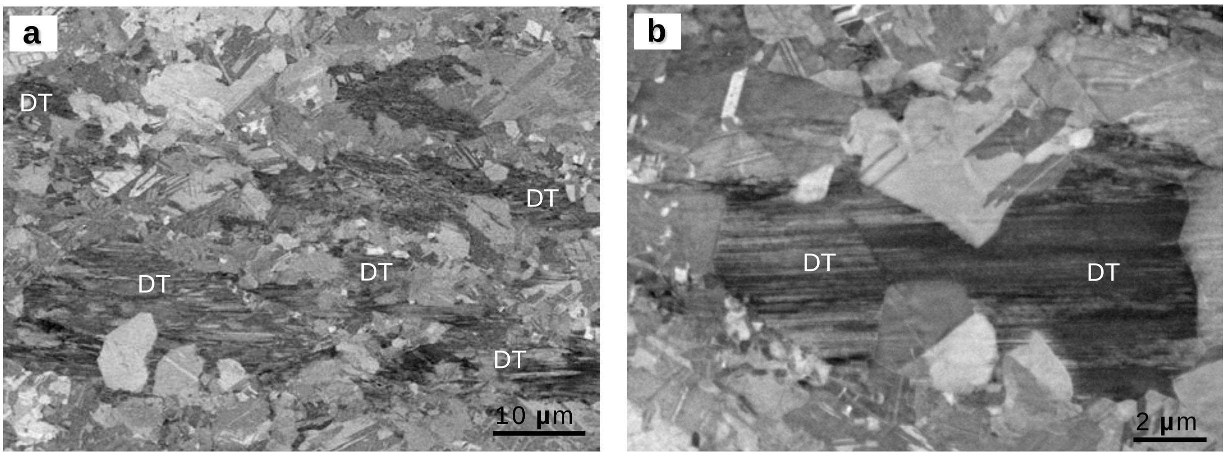

Fig. 3. SEM-ECC observations of Cu-4.5 wt.% Al samples processed by DPD to ε=1.7 and subsequently annealed at 300 ^{o}C for 20 minutes. (a) The overall microstructure; (b) a closer observation. "DT" indicates regions with high density of deformation twins.

Figure 4 shows the tensile behavior of the samples processed by DPD to ε=1.7 and subsequent annealing. The DPD treatment significantly increases the strength, but results in a very limited uniform elongation (ε_u=~0.01). Such an effect of strengthening accompanied by a loss of ductility is typical of nano-structured materials (Meyers, Mishra and Benson 2006). Annealing of the DPD samples in general leads to a reduction of the strength and an increase of ε_u. However, no substantial improvement of ε_u was obtained when the annealing temperature is below 270 ^{o}C (f_{RX}=34.2%), although the flow stress significantly dropped. An obvious increase in ε_u is obtained only when the annealing temperature exceeds 280 ^{o}C (f_{RX}=69.0%). The flow stress at a true strain of 2%, $\sigma_{2\%}$, and ε_u of the DPD samples annealed at various temperatures for 20 minutes, as a function of f_{RX}, are plotted in Fig.4b and c, respectively. Note that in the present work, $\sigma_{2\%}$ is used instead of the yield stress, $\sigma_{0.2\%}$, due to the relatively large uncertainty in determining $\sigma_{0.2\%}$ for tensile curves of soft materials which do not have a well-defined yield point.

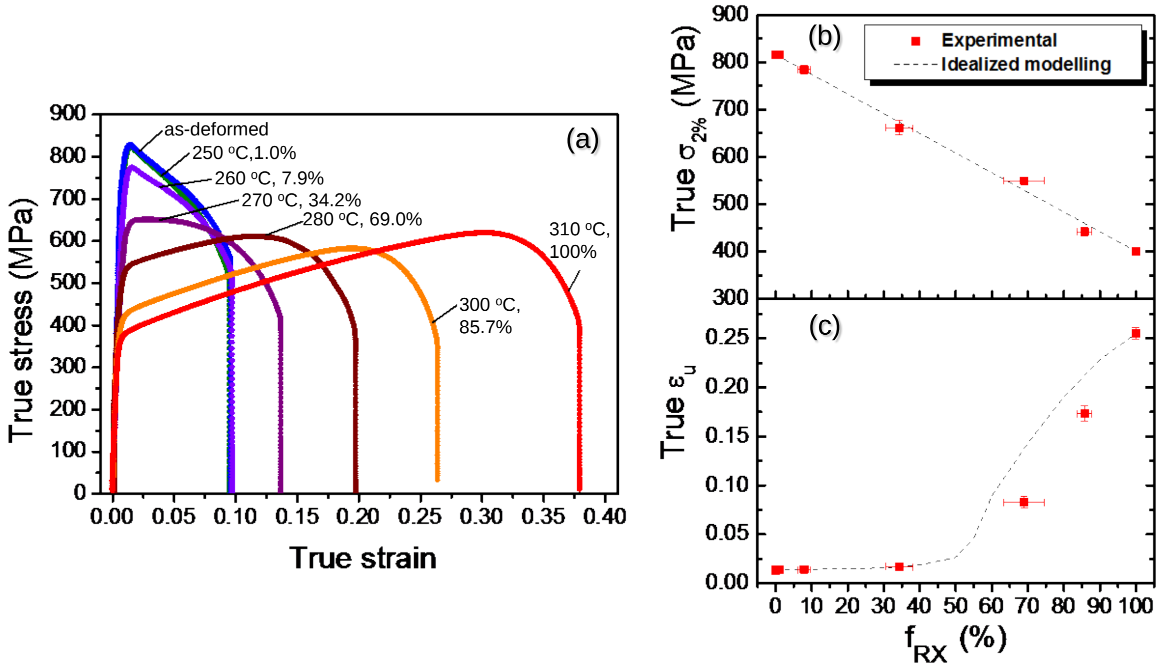

Fig. 4. (a) Tensile true stress-strain curves of Cu-4.5 wt.% Al samples processed by DPD to ε=1.7 and subsequently annealed at indicated temperatures for 20 minutes. The volume fraction of recrystallized grains is marked beside the annealing temperature; (b & c) the flow stress at a true strain of 2%, $\sigma_{2\%}$, and ε_u as a function of recrystallized volume fraction, f_{RX}. The experimental results are marked by red. The dashed curves are model results (see Section 4).

4. IDEALIZED MODELLING

An analysis of the tensile stress-strain behavior of a partially recrystallized sample requires knowledge of 1) the tensile stress-strain behavior of the non-recrystallized regions, 2) the tensile behavior of the recrystallized regions, and 3) the local stress and strain distributions of the different regions during tensile deformation, and experimental measurement of these parameters is a challenge. It has therefore been chosen to analyze the tensile behavior of a partially recrystallized sample by modelling based on a few assumptions.

For modelling of the flow stress of partially recrystallized samples, the following assumptions are the basis:

(1) The flow stress of the non-recrystallized regions is identical to that of the as-deformed samples up to the strain corresponding to the ultimate tensile strength, and at larger strains the work hardening is negligible and the flow stress is assumed to be constant, as plotted as a blue line in Fig. 5. This assumption is based on the observations that the T/M lamellae show little change and that the high density of dislocations are preserved after annealing at up to 300 ^{o}C for 20 minutes, both of which suggest little recovery (softening) of the non-recrystallized regions.

(2) The flow stress of the recrystallized regions is identical to that of the fully recrystallized samples annealed at 310 ^{o}C for 20 minutes (Fig. 5).

(3) Isostrain condition, i.e. the strain of the recrystallized regions and that of the non-recrystallized regions are both identical to the external strain during tensile deformation.

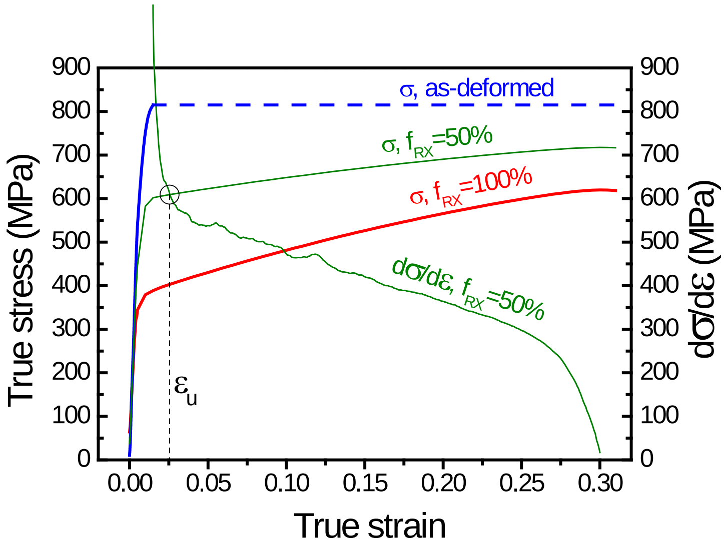

Fig. 5 An example to show modelling of the tensile behavior for partially recrystallized samples with f_{RX}=50%. Blue line: assumed tensile curve of the non-recrystallized regions based on the tensile curve of the as-deformed samples. Red line: assumed tensile curve of the recrystallized regions using the tensile curve of the fully recrystallized sample. Green line: modelled flow stress and work hardening rate of the samples with f_{RX}=50%. The intersection between the flow stress curve and the work hardening rate, as circled, corresponds to the uniform strain, ε_u.

Based on these idealizations, the flow stress of a partially recrystallized sample at a given strain is obtained according to the rule of mixtures: $\sigma=\sigma_{DPD}*(1-f_{RX})+\sigma_{RX100}*f_{RX}$, where σ is the flow stress of the entire sample, σ_{DPD} is the flow stress of the as-deformed sample, σ_{RX100} is the flow stress of the fully recrystallized sample. Once the tensile stress-strain curve is obtained, its work hardening rate can be found by simply differentiating the curve. The uniform strain can be determined using the Considére criterion (Hart 1967), i.e. by finding the strain where $\Theta=\sigma$, where Θ is the work hardening rate. In practice, ε_u is determined by finding the intersection between the flow stress curve and the work hardening rate curve. The derivation of the flow stress, work hardening rate and uniform strain for a partially recrystallized sample with f_{RX}=50% is shown in Fig. 5 as an example.

The rule of mixtures has been used in partially recrystallized materials for evaluation of the flow stress (Hansen & Vandermeer, 2005). In a previous study, Mileiko (Mileiko 1969) evaluated the strength and ductility of continuous fiber composites using the rule of mixtures in the isostrain condition. In the present work, the work hardening rate is calculated directly from the experimental tensile curve for better precision instead of using a fitted tensile curve as in Mileiko’s work.

The model-predicted flow stress curves and work hardening rate curves are shown in Fig.6a and b, respectively of the partially recrystallized samples with f_{RX}=34.2%, 69.0%, and 85.7% in comparison with the experimental curves. The $\sigma_{2\%}$ and ε_u obtained by the model for the partially recrystallized samples as a function of f_{RX} are plotted in Fig. 4b and c, respectively. In general the idealized model gives rather good prediction of the flow stress (Fig. 4b and 6a). The modelled uniform strain as a function of f_{RX} shows a similar trend as the experimental results – the uniform strain only increases significantly when f_{RX} exceeds a certain amount in the range of

45-65%. The prediction of the work hardening rate (Fig. 6b) and the uniform strain (Fig. 4c) is less precise compared with the prediction of the flow stress. As shown in Fig. 6b, for the samples with f_{RX}=69.0% and 85.7%, although the predicted work hardening rate curves of the two samples are comparable with the experimental results, the experimental results are larger at low strains and decrease faster with increasing strain compared with the model results. This leads to a certain over-prediction of ε_u for these two samples (Fig. 4c). This discrepancy is however acceptable as the relationship between structural parameters and elongation is more uncertain than that between structural parameters and strength.

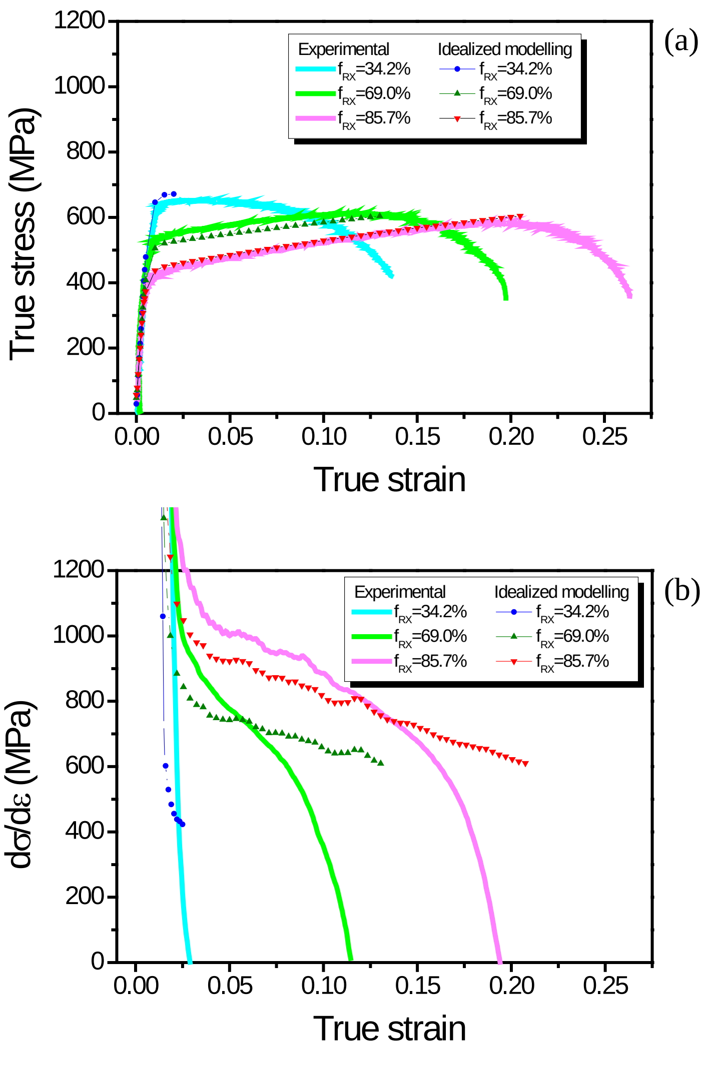

Fig. 6 Comparison between experimental results and model predictions of (a) flow stress curves and (b) work hardening rate curves for Cu-4.5 wt.% Al samples recrystallized to different f_{RX} as indicated.

5. DISCUSSION

5.1 Linear relation between the flow stress and f_{RX}. In the idealized model, a linear relation between the flow stress and f_{RX} must be expected for partially recrystallized samples (Fig. 4b). The good agreement between the experimental measurements and model predictions suggest that the idealization of flow stresses in the model is satisfactory, especially the assumption that there is very limited recovery in the non-recrystallized region, which is supported by the small change in the T/M lamellar thickness in the DPD samples after annealing.

5.2 Nonlinear relation between ε_u and f_{RX}. In contrast to the flow stress, the ε_u of a composite is not directly additive, and the determination of ε_u requires the Considére criterion being applied. As in Fig. 4c, a nonlinear relation between ε_u and f_{RX} is characteristic of an idealized model, which is also observed from the experimental results. According to the model, ε_u shows little increase at low f_{RX} due to the work hardening rate being insufficient compared with the high flow stress. The example in Fig. 5 shows that although the work hardening rate is increased substantially in the samples with f_{RX}=50% compared with the as-deformed samples, ε_u remains small because of the work hardening rate being smaller than the flow stress.

The model also suggests that for a partially recrystallized material, a smaller difference in the strengths between the non-recrystallized regions and the recrystallized regions, and/or a higher work hardening rate of the different regions, are beneficial for increasing ε_u at low f_{RX}. In partially recrystallized Cu processed by cold rolling with subsequent annealing, ε_u starts to increase significantly when f_{RX} exceeds 20% (Lin, Delannay, Zhang, Pantleon and Juul Jensen 2014).

6. CONCLUSIONS

Microstructural characterization and tensile tests show that the flow stress of partially recrystallized Cu-4.5 wt.% Al decreases approximately linearly as a function of f_{RX}, whereas ε_u only starts to increase until f_{RX} exceeds a certain value in the range of 45-65%.

By assuming that the hard component has an identical property to the as-deformed sample, that the soft component has an identical property of the fully recrystallized samples, and that the two components fulfil the isostrain condition, the idealized model gives a rather accurate approximation to the flow stress, and a less accurate approximation to the work hardening rate (and thus ε_u) of the partially recrystallized samples. Compared with the model predictions, the experimental work hardening rates are higher at low strains, but decrease faster as the strain increases. According to the model, the linear decrease of flow stress and the nonlinear increase of ε_u as a function f_{RX} are characteristics of a composite that fulfils the model’s assumptions.

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REFERENCES

Hansen, N. and Vandermeer, R. (2005). Recovery, recrystallization, and grain growth. In F. Bassani, J. Liedl and P. Wyder (Eds.), Encyclopedia of condensed matter physics (pp. 5:116–124). Elsevier.

Hart, E. . (1967). Theory of the tensile test. Acta Metallurgica, 15, 351–355.

Huang, X., Hansen, N. and Tsuji, N. (2006). Hardening by annealing and softening by deformation in nanostructured metals. Science (New York, N.Y.), 312, 249–51.

Koch, C. C., Morris, D., Lu, K. and Inoue, A. (1999). Ductility of nanostructured materials. MRS Bulletin, 24, 54 – 57.

Li, Y., Zhang, Y., Tao, N. and Lu, K. (2008). Effect of thermal annealing on mechanical properties of a nanostructured copper prepared by means of dynamic plastic deformation. Scripta Materialia, 59, 475–478.

Lin, F. X., Delannay, L., Zhang, Y. B., Pantleon, W. and Juul Jensen, D. (2014). Partially recrystallized microstructures of cold-rolled copper and mechanical properties. In These proceedings.

Meyers, M. A., Mishra, A. and Benson, D. J. (2006). Mechanical properties of nanocrystalline materials. Progress in Materials Science, 51, 427–556.

Mileiko, S. (1969). The Tensile Strength and Ductility of Continuous Fibre Composites. Journal of Materials Science, 4, 974–977.

Murr, L. (1975). Interfacial Phenomena in Metals and Alloys (pp. 87–164). Addison-Wesley Publishing Company.

Tsuji, N., Ito, Y., Saito, Y. and Minamino, Y. (2002). Strength and ductility of ultrafine grained aluminum and iron produced by ARB and annealing. Scripta Materialia, 47, 893–899.

Wang, H. T., Tao, N. R. and Lu, K. (2012). Strengthening an austenitic Fe–Mn steel using nanotwinned austenitic grains. Acta Materialia, 60, 4027–4040.

Wang, Y., Chen, M., Zhou, F. and Ma, E. (2002). High tensile ductility in a nanostructured metal. Nature, 419, 912–5.

Yan, F. K., Liu, G. Z., Tao, N. R. and Lu, K. (2012). Strength and ductility of 316L austenitic stainless steel strengthened by nano-scale twin bundles. Acta Materialia, 60, 1059–1071.

Zhao, Y., Topping, T., Bingert, J. F., Thornton, J. J., Dangelewicz, A. M., Li, Y., Liu, W., Zhu, Y.T., Zhou, Y.Z. and Lavernia, E. J. (2008). High Tensile Ductility and Strength in Bulk Nanostructured Nickel. Advanced Materials, 20, 3028–3033.