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Evolution of microstructure and crystallographic texture in severely cold rolled high entropy equiatomic CoCrFeMnNi alloy during annealing

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Abstract. An equiatomic FCC CoCrFeMnNi high entropy alloy (HEA) was heavily cold rolled up to 90% reduction in thickness followed by isochronal annealing for 1 hour at temperatures ranging between 700°C to 1100°C. A strong brass texture was observed in the cold-rolled condition indicating the low stacking fault energy of the material. A fine stable microstructure was observed during annealing at low temperatures. The recrystallization texture was characterized by the presence of deformation texture components, in particular, the α-fiber (ND//<110>), S ({123} <634>) and the typical brass recrystallization texture component ({236} <385>). Annealing twins were shown to have important effect on the formation of annealing texture.

1. Introduction

High entropy alloys (HEAs) are recently developed multicomponent alloys consisting of at least five or more elements in equimolar or near equimolar proportion but with simple crystal structures e.g. FCC, BCC or FCC+BCC [1]. The retention of simple crystal structure instead of formation of complex intermetallic phases is attributed to large configurational entropy due to mixing of a large number of elements [1]. Recently, many multicomponent alloy systems with simple crystal structures have been proposed. Amongst these FCC CoCrFeMnNi alloy has attracted much attention due to excellent thermal stability, good ductility and other attractive properties [2-3].

Recent studies show that the single phase structure is stable in this alloy even after annealing for 72h at 1000°C [4]. It is expected that appropriate thermo mechanical processing can greatly enhance the properties of these alloys. However, this necessitates appropriate understanding of microstructure and texture evolution during thermo-mechanical processing in these alloys. In the present work effect of heavy cold-rolling and annealing on microstructure and texture evolution in CoCrFeMnNi is investigated.

2. Experimental procedure

An equiatomic CoCrFeMnNi alloy was used in the present study. As received block having dimensions 50 mm (length) × 8 mm (width) × 8 mm (thickness) were produced by vacuum arc melting. To enhance the chemical homogeneity the blocks were homogenized for 6 h at 1100°C. In order to breakdown the coarse microstructure (average grain size ~200 μm), samples of ~25 mm (length) × 8 mm (width) × 5 mm (thickness) were cut from the blocks and then cold rolled to ~ 50% reduction in thickness using a laboratory scale rolling mill followed by annealing at 800°C for 1 h in a salt bath furnace. The fully recrystallized samples with thickness of 2.5 mm were used as starting material. The grain size of starting material was ~8 μm. The starting materials were cold-rolled to ~90% reduction in thickness to a final thickness of 0.25 mm.

The microstructure and microtexture of the deformed and annealed samples were investigated by Electron Back Scatter Diffraction (EBSD) unit attached to a FE-SEM (Supra 40; Carl-Zeiss, Germany) operated at 20kv. The EBSD measurements were taken from ND-RD plane for 90% cold rolled samples.

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For annealed samples the scans were taken in both ND-RD and rolling planes (RD–TD plane). The volume fraction of individual texture components were calculated using a cut off angle of 15° around the ideal locations in the Euler space. TSL-OIM^{TM} software was used for an analysis of the acquired EBSD data.The samples were prepared by mechanical polishing followed by electropolishing using a mixture of methanol and percholoric acid (9:1 by volume).

3. Results and discussion

Figure 1 (a) shows the grain boundary (GB) map of starting material. The GB map clearly shows the typical microstructure of fully recrystallized material. The average grain size of the starting material is ~8 µm (excluding the $\Sigma3$ boundaries). Figure 1 (b) shows the pole figure (PF) of the starting material which indicates the presence of rather weak texture.

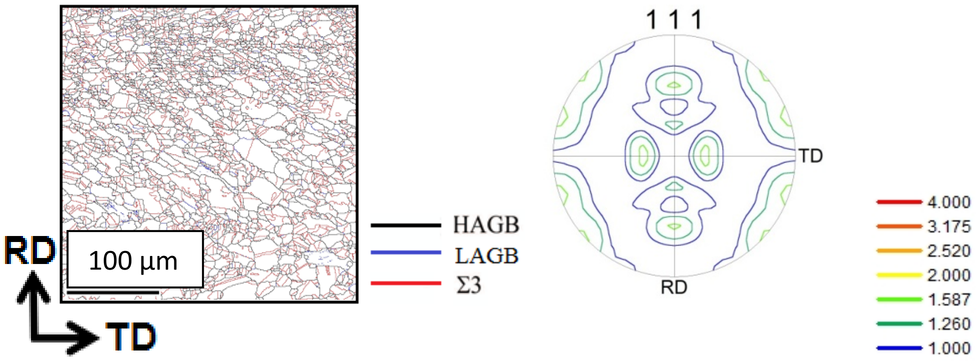
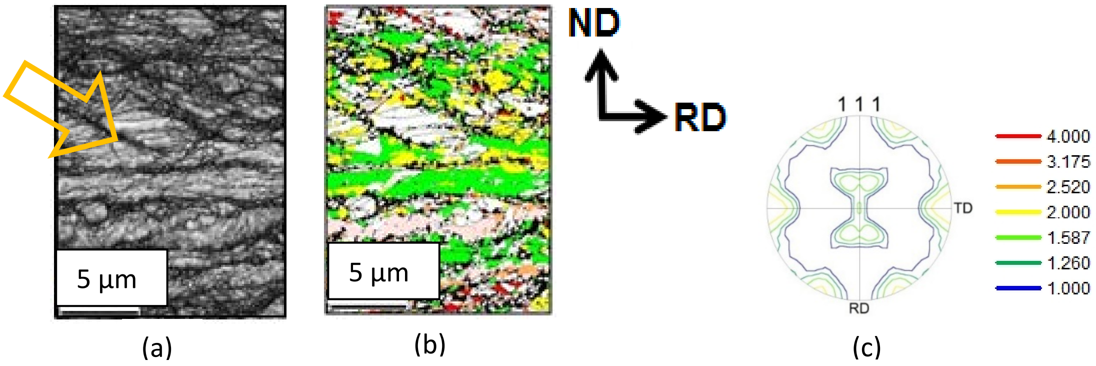


Fig. 1: (a) Grain boundary map and (b) (111) PF of the starting material.

Microstructure and texture of the severely deformed material

The image quality map clearly shows the heavily fragmented structure and thin shear band also observed in the microstructure (~25° to rolling direction indicated by arrow mark). Fig 2(b) and (c) show the grain orientation map (GO Map) and pole figure of the 90% cold-rolled sample. It shows the strong brass (green) texture indicating low stacking fault energy this alloy [5].



(a) (b) (c)

C	Cu	S	B$_S$	G	G/B	A	BR
{001}<100>	{112}<111>	{123}<634>	{110}<112>	{110}<001>	{110}<115>	{110}<111>	{236}<385>
🟦	🟥	🟨	🟩	🟧	▨	🟩	🟪

Fig. 2: (a) IQ map, (b) GO map and (c) (111) PF of 90% cold rolled material.

Microstructure and texture of the annealed material

Figures 3(a) and (b) show the grain orientation (GO) maps obtained from the RD-TD and ND-RD planes of the 700°C material, respectively. The GO maps obtained from rolling and ND-RD planes show fully recrystallized microstructure with average grain size 1.4μm and 1.1μm, respectively. The appearance of the (111) pole figures (Figures 3(c) and 3(d)) suggests presence of α-fiber component, namely, Brass/Bs (green), G/B (deep rose; {011}<115>)) and (A ($(110)[1\bar{1}1]$). Presence of S (yellow) is also noticed. Significant presence of the brass recrystallization component (BR) {236} <3 8 -5> is observed. A very similar average grain size ~34 μm is obtained from the RD-TD (Fig.4 (a)) and ND-RD planes (Fig.4 (b)), respectively after annealing at 1100°C. The GO maps also show the presence of very similar texture components observed in case of the material annealed at 700°C. The PFs do not reveal any substantial change in texture (Fig. 4 (c) and (d)).

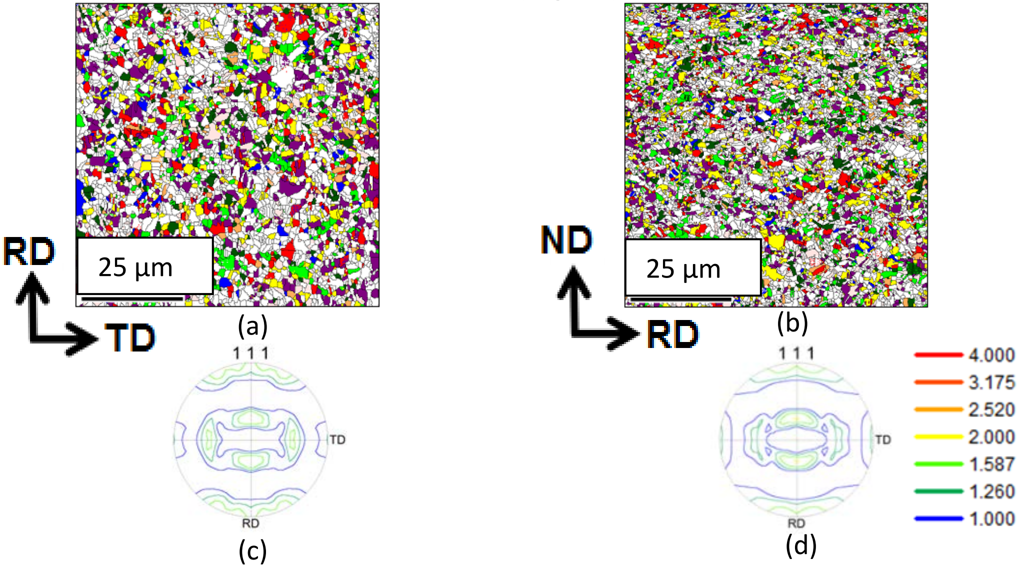


Fig. 3: GB maps (a, b) and (111) PFs (c, d) of the material annealed at 700°C obtained from the RD-TD (a,c) and ND-RD (b,d) planes. Color code is same as in Fig.2.

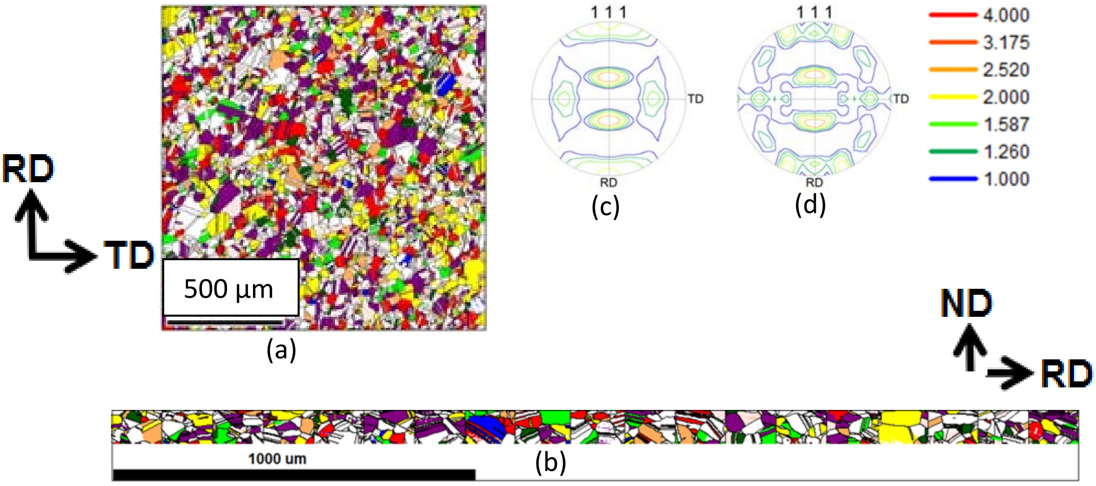


Fig. 4: GB maps (a, b) and (111) PFs (c, d) of the material annealed at 1100°C obtained from the RD-TD (a,c) and ND-RD (b,d) planes. Color code is same as in Fig.2.

The retention of deformation texture components is explained by the mechanism of discontinuous recrystallization without preferential orientation selection [6]. The development of α – fiber components, such as, A is explained by the formation of annealing twins [6]. The present analysis shows significant presence of the BR component in heavily deformed and annealed FCC HEAs.
The through-thickness homogeneity in texture of materials annealed at 700°C (Fig.5 (a)) and 1100°C (Fig.5 (b)) is shown quantitatively in Fig.5. Remarkable through-thickness homogeneity in texture is obtained after annealing at different temperatures.

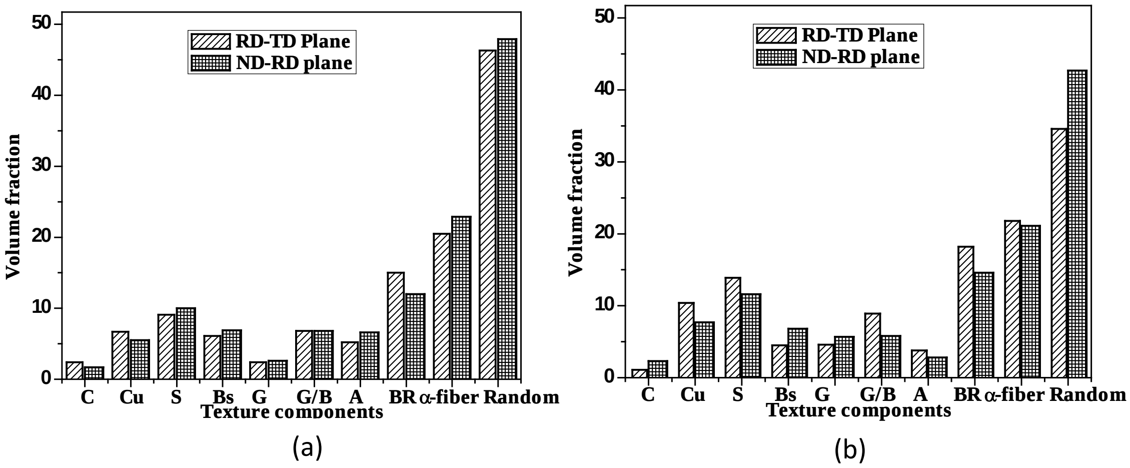


Fig. 5: (a) and (b) show the individual texture components in materials annealed at 700°C and 1100°C, respectively.

4. Conclusions

- Development of heavily fragmented microstructure after severe cold rolling is observed. The development of the strong brass type texture indicates the low stacking fault energy of this alloy.
- Ultrafine grain structure with mean grain size of 1.1 μm was obtained through conventional cold rolling and subsequent annealing at 700°C.
- The recrystallization texture has significant presence of the α – fiber components, S and BR components.
- Remarkable through-thickness homogeneity of microstructure and texture is obtained after annealing.

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