Microstructure evolution of C-doped high entropy alloys

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1. Introduction

The Cantor CoCrFeMnNi high entropy alloys and its family alloys are well-known multi-component alloys and drawing significant attraction due to their excellent strain hardening capability and high corrosion resistance. However, their strengths are as low as between 150-190 MPa of yield strength after solution treatment. In this study, the carbon is added to FCC solid solution CoCrFeNi high entropy alloy (MEA) for improving the strength due to more pronounce lattice distortion, interstitial solid solution hardening and precipitation hardening. Apart from the enhanced mechanical properties, the microstructure formation mechanism is also changed because of changed stacking fault energy and added interstitial carbon atoms. Therefore, in this study, the microstructure evolution during annealing at 700°C - 1000°C was studied by in-situ neutron diffraction experiment in terms of dislocation density, fraction of carbide precipitation and texture evolution.

2. Experiment

An ingot of 24.72Co-24.72Cr-24.72Fe-24.72Ni-1C (at%) alloy was cast by using a vacuum induction furnace. As-cast samples were homogenized at 1200°C for 5 h. A rectangular section (25 × 40 × 100 mm3) of homogenized samples were cold rolled by 74% total thickness reduction until 6.6 mm thickness of the sheet. After cold rolling, the sticks (52 x 6 x 2 mm3) were prepared for in-situ heating neutron diffraction experiment at 700°C, 800°C, 900°C and 1000°C for 60 min.. The neutron diffraction experiment was performed in-situ during heating at the Time-of-Flight (TOF) neutron diffractometer, BL20 iMATERIA, at J-PARC MLF, Ibaraki, Japan. For data analysis, the acquired neutron diffraction data was divided into a 180 s acquisition time increment. The dislocation density was evaluated by using CMWP method. The phase identification and crystallographic texture were analyzed by using Rietveld analysis with MAUD software on the neutron diffraction data.

3. Results

After 74% cold rolling, the inhomogeneous deformation and strong grain refinement led to dislocation density as large as 1.6 x 10^{16} m^{-2}. The dislocation rearrangement parameter or *M* parameter was characterized as of 0.3, indicating strong dislocation rearrangement and a formation of dislocation cell wall. The double α <110>//ND and γ <111>//ND fiber textures were presented after 74% thickness reduction of cold rolling.

Apart from FCC solid solution phase, the $M_{23}C_6$ carbide was characterized by neutron diffraction patterns during the annealing. Overall, $M_{23}C_6$ content increased with annealing temperature and time. After annealing, the deformation textures including the double fiber textures became weaker with an increase in annealing time and temperature. Interestingly, the retentions of deformation texture were observed in the ODFs of the samples annealed at 700°C, 800°C and 900°C. In contrast, the texture randomization was developed at early 1000°C annealing. The chain-like carbides were formed during the annealing at 700°C, 800°C and 900°C. In contrast, the larger and randomly

distributed carbides were observed during annealing at 1000°C. It can be suggested that the formation characteristic of carbide precipitation were correlated to the microstructure and texture evolution.

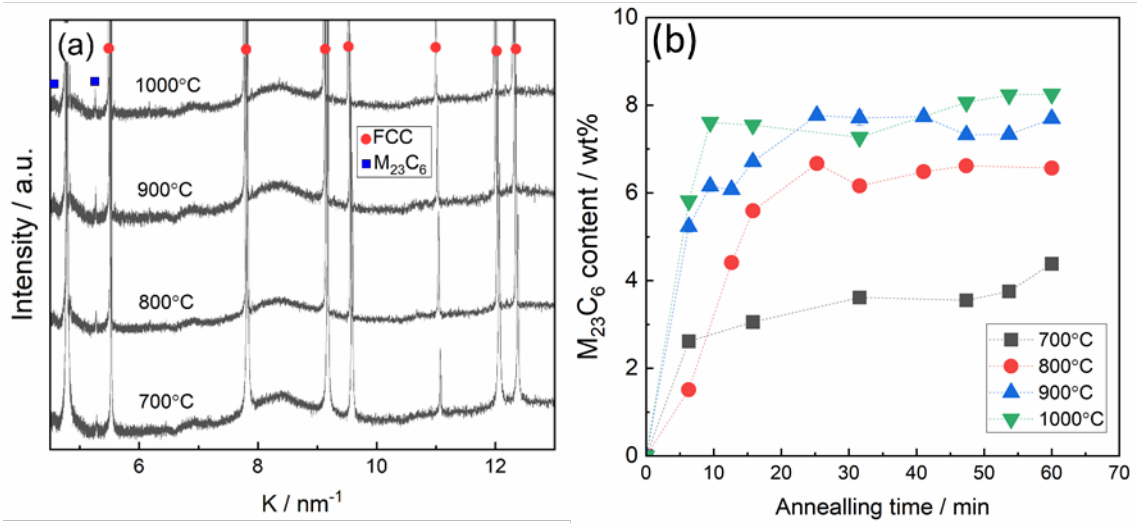

Fig. 6 (a) Fig. 1 Neutron diffraction pattern of the samples after 60 minutes annealing, and (b) an increase of M23C6 content during annealing at 700°C, 800°C, 900°C and 1000°C.

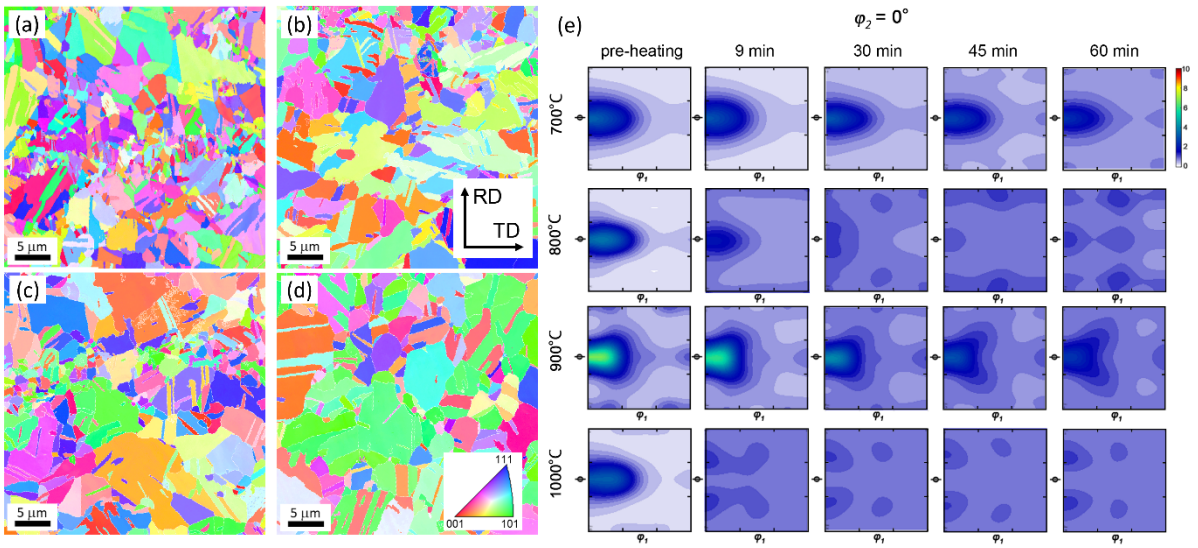

Fig. 3 IPF maps of the samples annealed at (a) 700°C, (b) 800°C, (c) 900°C and (d) 1000°C for 60 mins and (e) φ2 = 0° sections of ODFs calculated from neutron diffraction data of 1%C-doped CoCrFeNi alloy during the annealing.

4. Conclusion

The microstructure evolution of 74% rolled 1%C-dope CoCrFeNi HEA was studied by using in-situ heating neutron diffraction experiment at 600°C, 700°C, 800°C and 900°C. The line profile analysis revealed quite high dislocation density after rolling, which indicated a high stored strain energy was introduced. The carbides was increasingly precipitated with annealing temperature and time. The carbide morphology and characteristic were observed to correlate to the microstructure and texture evolutions.