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利用課題名（日本語）　：X 線回析による金属材料の弾塑性変形挙動の解析
Program Title (English)　：Elastic and plastic behavior of alloys investigated by synchrotron X-ray diffraction
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1. 概要（Summary ）

High entropy alloys (HEAs), which consist of multi-principal metallic elements, have attracted extensive academic interests. Amongst, face-centered cubic (fcc) phase FeMnCoCrNi alloy is one of the most successful HEAs, exhibiting excellent tensile strength and ductility at both ambient and cryogenic temperatures [1,2]. The exceptional mechanical property is due to their low stacking fault energy (SFE), in which the deformation twinning (DT) contributed to the plasticity. Moreover, we recently designed strong and ductile novel Co-rich HEAs by further reducing SFE, which exhibits DT and fcc → hcp martensitic transformation (MT) behavior [3-5]. Both of the DT and MT are progressed by the glide of Shockley partial dislocations on the {111} slip planes. On the other hand, the spatial composition variation or undulation is appreciable and normal. There is a local preference of bonding among certain nearest-neighbor atoms leading to form short-range order (SRO), which could be tuned by modifying the compositions and heat treatments.

However, the influence of the regulated-SRO on the plasticity of HEAs is unclear. Moreover, the effect of SRO on the mechanical twinning and fcc → hcp SIMT has not been declared. In the fcc-metals with low SFE, one perfect dislocation will dissociate into a pair of Shockley partials bonded to a stacking fault. The glid of partial dislocations on consecutive {111} planes forms a nano-twin, whereas the glid on every second {111} planes produces the hcp structure.

In the present study, we conducted *in-situ* deformation measurement for revealing the defects evolution kinetics in various high entropy alloys. We aimed to clarify the influence of SRO on the multiscale plasticity behavior of HEAs, particularly the nucleation and propagation of dislocations, the DT, and the MT in the HEAs. This is pursued by investigating the micro-plastic deformation behavior of the HEAs using the *in-situ* high-energy synchrotron X-ray diffraction method, in which the evolution of microstructures was observed and the plasticity constitutive relationships was clarified.

2. 実験（目的,方法）（Experimental）

Purpose: The main purpose of the proposed study is to measure the microstructural evolution of high entropy alloys during tensile deformation, for determining the dislocations, stacking faults, and/or phase-transformation quantitatively. We utilized the high energy synchrotron X-ray diffraction, which is one of the most promising techniques for this measurement. It gives the best statistical measurement by interrogating a large three-dimensional volume. The averaged information such as dislocation density, stacking fault probability, phase volume can be accurately tracked in real-time corresponding to the applied stress or strain.

Method: In the experiment, the typical SRO-regulated HEAs based on our high-throughput *ab initio* calculations and Monte Carlo simulations were selected for experiments. The samples were prepared by arc-melting, hot-forging, cold-rolling, and annealing to obtain fully-recrystallized structures with refined grain structures (space group: $Fm\bar{3}m$, average grain size <10μm). After that, tensile samples with a thickness of ~0.5 mm were prepared for conducting the *in-situ* X-ray diffraction measurement using the beamline BL22XU of JAEA at SPring-8. The incident X-ray beam size was 0.2 mm × 0.2 mm, and the diffraction patterns will be collected using a two-dimensional large-area pixel detector (PILATUS-100K, DECTORIS). The X-ray beam energy was 30 keV. The strain rate for tensile deformation is 1.0×10^{-3} s^{-1}, and diffraction patterns under various strain levels were recorded. The dislocation density, stacking fault probability, and volume fraction of the hcp phase in the tensile deformed SRO-regulated HEAs were analyzed from diffraction profiles, in which the diffraction peak shift, broadening, and the peak intensity change were measured. The strengthening and hardening behavior will be analyzed and compared thoroughly, in which the crystal plasticity constitutive relations based on the measured parameters were clarified. The following methods will be utilized for analyzing the diffraction profile: 1. the single peak fitting will be done by using Z-Rietveld code. 2. The Rietveld Refinement of the diffraction profile is conducted by using Material Analysis Using Diffraction (MAUD) software. 3. The Convolutional Multiple Whole Profile fitting (CMWP) method is utilized for quantitative determining the dislocation density and stacking fault probability.

3. 結果と考察（Results and Discussion）

The in-situ tests were conducted, and the 2D-diffraction patterns were acquired successfully in real-time as shown in Fig. 1. The diffraction profiles were obtained from the analysis of the 2D images. As can be seen, the peak of (111) plane of FCC-phase tends to become broadened during

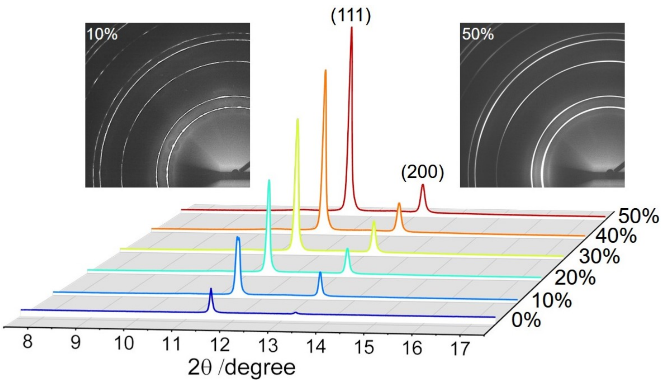

Fig. 1. The X-ray diffraction pattern profiles and images of the DT-HEA, which were acuqired when the samples were tensile to an engineering strain of 0%, 10%, 20%, 30%, 40%, and 50%, respectively.

plastic deformation, due to the multiplication and storage of dislocations and stacking faults. Moreover, the intensity becomes stronger with the increase of strain, which indicates that the grains were rotated along tensile direction. This leads to the formation of strong fiber texture in the samples. Noting that deformation-induced new phase was not observed in the HEA, i.e., strain-induced martensitic phase transformation from FCC-phase to HCP-phase was not proceeded during tensile deformation. Based on the results, one can conclude that the plastic deformation of the samples is accomplished by dislocation slips and stacking faulting. A fiber (111) texture was formed by tensile deformation. The defects evolutions will be quantitatively analyzed and the constitutive relationships will be constructed.

4. その他・特記事項（Others）

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