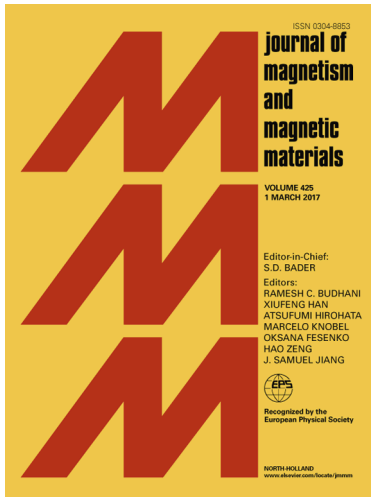
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Preparation of Magnetic Fe-Co Alloy Using Texture Controlled technique

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Abstract

Microstructure and texture controlled 48Fe-52Co at% alloy was prepared by repeated cold-rolling and recrystallization through heat treatment at temperatures below order-disorder phase transformation using Fe and Co foils. Furthermore, distortion of controlled texture orientated cubic crystal structured 48Fe-52Co at% alloy along the <001> direction was attempted by introducing nitrogen interstitially through plasma nitriding. The textural analysis suggested that the microstructure is composed of micron-sized grains randomly aligned along rolling direction (RD). Furthermore, from the results of orientation distribution function (ODF), the dominance γ-fiber was confirmed in as-rolled samples. However, in recrystallized sample, the intensity of rotated-cube orientation increased while the presence of γ-fiber decreased. Furthermore, a complete rotated-cube orientation was realized in nitrided texture. The appearance of the prominent X-ray diffraction peak (200) in all samples indicated the presence of rotated-cube orientation. However, the FWHM of (200) peak of the nitrided sample was broader than the rest. This was attributed to the diffusion of nitrogen into the matrix during plasma nitriding and internal nitriding was confirmed from the EDX analysis. However, further investigation is necessary to understand the behavior of nitrogen.

Magnetic properties of as-rolled 48Fe-52Co at% alloy inferred from in-plane magnetic hysteresis loops was similar Fe foil. Furthermore, magnetic properties of recrystallized and nitrided 48Fe-52Co at% alloy were different from Fe-Co particles or crushed bulk Fe-Co alloy prepared by arc melting. However, the internal diffusion of nitrogen was not adequate enough to bring about a considerable change in the magnetic properties. We believe that considerable crystal distortion to induce change in the magnetic properties can be exerted by introducing interstitial carbon and the addition of third or fourth elements.

Keywords: *Fe-Co, cold rolling, mechanical alloying, microstructure, rotated-cube orientation, magnetic property*

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1. Introduction

Metallic materials with diverse functional properties have been developed and used in many engineering applications. These materials are fabricated by using metal processing techniques such as melting and mixing of metals using arc or high-frequency induction melting furnaces, forging, press working, powder metallurgy, casting etc. Among them, particular interest is vested in the method used to fabricate metallic materials using cold-rolling and recrystallization called "texture control" technique. The texture controlled metallic materials are known to improve the moldability, high tensile strength and magnetic property. Consequently, this technique is being extensively used in a wide range of commercial application for the fabrication of thin steel sheets for the external body plate of automobiles.

We have been working on the development of functional metallic materials such as hydrogen storage alloys [1-5] and magnetic recording materials [6], using the "texture control" technique. We also succeeded in establishing the super-lamination technique using repeated cold-rolling [7].

Fe and Co are well-known and abundantly available resources that are being widely used in various applications. Fe-Co alloy is a soft magnetic material and the high magnetic permeability alloy, $Fe_{50}Co_{50,}$ which is called "Permendule", is being used in the fabrication of magnetic lens, motor, magnetic valve, electromagnet, etc. [8]. Recently, a very interesting study based on the first principle calculation by Burkert et al. [9] proposed the use of Fe-Co alloy for hard magnetic material applications. According to their calculation, if the B2(CsCl type) crystal structured Fe-Co is strained or distorted along the c-axis and if the c/a ratio assumes a value between 1.20 and 1.25, they begin to possess hard magnetic properties with very high coercivity, suitable for permanent magnet applications.

Deng et al. have reported that when the texture controlled Fe-Co alloy is heat treated below the order-disorder transformation temperature of 730 °C, rotated-cube is oriented in {100} <011> [10-11]. On the other hand, in the case of Fe-Si alloy, the crystal is orientated in Goss (easy magnetization direction) {011} < 001 > and Cube {100} < 001 > directions. Rotated-cube orientation of {100} < 011 > is obtained by rotating the cube orientated crystal by 45 degrees. However, the above study was limited to texture control and the magnetic properties were not reported. In addition, textured materials with their magnetic easy axis oriented in the Goss and Cube orientations are being used as magnetic steel plates in various engineering applications. On the other hand, Ahmad et al. have investigated the texture development and magnetic properties of dual phase Fe-Co-X alloys prepared under different degrees of cold rolling and heat treatment conditions [12-16].

In this study, the application of super-lamination technique on the development of structure and texture controlled magnetic 48Fe-52Co at% alloy is investigated. And also, the possibility introducing strain to the cubic structure and their subsequent influence on the magnetic properties was evaluated by nitriding the 48Fe-52Co at% alloy.

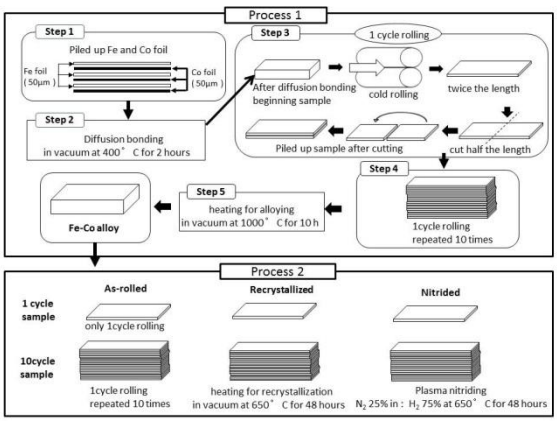


Fig . 1 Procedure for the preparation of as-rolled, recrystallized and, nitrided samples.

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2. Materials and methods

2.1. Preparation of cold rolled textures

Fig. 1 shows preparation of as-rolled recrystallized and nitrided samples. During process 1 ten pieces each of Fe and Co foil with dimensions 20 mm x 30 mm x 50 μm were piled up alternately (Step 1) and diffusion bonded by treating in the vacuum, at 400 °C for 2 hours (Step 2). Then, the specimen was cold-rolled repeatedly for 10 times using the laboratory rolling mill manufactured by YOSHIDA KINEN Co., Ltd. When the length of the sample became almost double the original length, it was cut into half and piled up one on top of the other. This process is defined as "one cycle" (Step 3). The cold rolled texture was prepared by carrying out ten cycles (Step 4). The chemical composition and thickness of the cold rolled sample were Fe-48%Co-52% (at.%) and approximately between 300 to 500 microns, respectively. Then, the sample was alloyed by heating in vacuum, at 1000 °C for 10 hours (Step 5) to obtain Fe-Co alloy. The alloyed sample was cold rolled for 1 cycle and 10 cycle to prepare control orientated or microstructure textured specimens. This sample is defined as "as-rolled sample". Recrystallized texture sample was obtained by annealing the as-rolled sample at 650 °C for 48 hours in vacuum [17-20]. Nitrided samples were prepared by plasma nitriding [21] as-rolled sample under N_2 25% in H_2 in reaction tube heated at 650 °C for 48 hours.

2.2. Characterization

The texture observation, crystal structure analysis, crystal orientation distribution function (ODF) and magnetic properties of (a) as-rolled, (b) recrystallized texture and (c) nitrided samples were carried out using SEM-EDX, XRD, ODF and VSM, respectively.

Microstructure and texture were observed by scanning electron microscope S-3200N Hitachi, Ltd. and elemental analysis was measured by Energy dispersive X-ray spectrometry EMAX-7000 by Horiba Ltd. For SEM-EDX measurements, the surface of specimen cross-sections was polished to mirror condition by mechanical polishing using abrasive paper and buff cloth using alumina powder.

Crystal structures analysis was carried out using X-ray diffractometer [Smart Lab by RIGAKU:] Cu Kα(wavelength: λ=0.15418nm), operating conditions: 40kV 40mA). And monochromater was used to reduce the florescence X-ray and the measurement mode was set to reduced florescence X-ray mode. Texture measurement was carried out using Schulz's reflection method, and by plotting pole figures of (1 1 0), (2 0 0), (2 1 1), (2 2 0), (2 2 2), (3 1 0). The orientation distribution function (ODF) was determined by using X'pert-MRD Philips X-ray diffractometer with a maximum tilt angle of 85 degrees in steps of 5 degrees: Cu Kα(λ= 0.15418nm), operating conditions: 50kV 40mA. ODF was made by the Euler angles (φ_1, Φ φ_2)

Electron Back-Scattering Diffraction (EBSD) pattern was obtained by Orientation Imaging Microscopy Crystal Orientation System (OIM) by TexSEM Laboratories, Inc. and Scanning Electron Microscope JSM-6390 JEOL. Magnetic properties were measured using TM-VSM 1230-HHH5 by Tamakawa Co. Ltd. at room temperature.

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3. Results and discussion

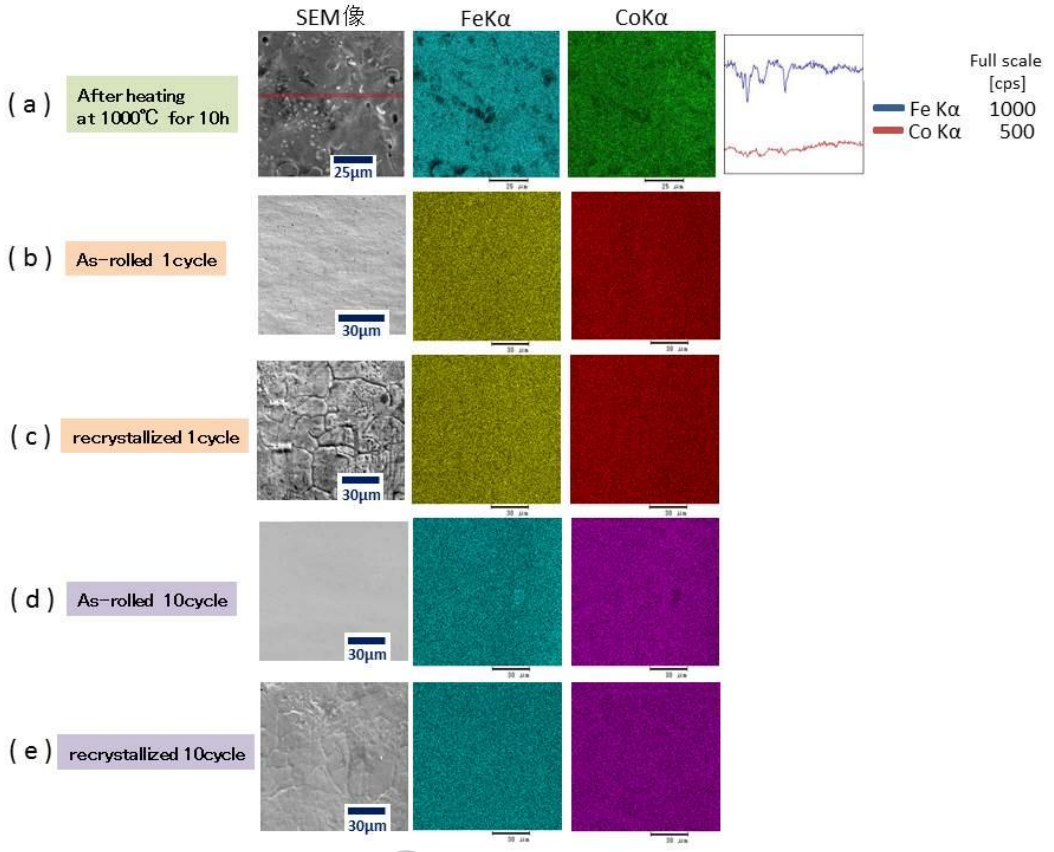


Fig. 2 SEM-EDX (a) after heating at 1000℃for 10h (process 1-step 5), (b) as-rolled (1 cycle) (c) recrystallized (1 cycle), (d) as-rolled (10 cycles) and (e) recrystallized 10cycles

Fig. 2 shows the SEM-EDX results of alloyed Fe-Co. In the case of Fig. 2(a) the sample underwent alloying and recrystallization due to the treatment carried out during step 5 of process 1. i.e.: treated at 1000°C・10h. Consequently, Fe-Co binary alloy with recrystallized texture is believed to have formed. Since, the above sample is cold rolled for 1 and 10 cycles in Process 2, Fe-Co binary alloy with rolling texture is formed as shown in Fig. 2 (b) ad (d). When this sample is annealed at 650°C・48h, Fe-Co binary alloy with recrystallized texture is believed to have formed in the case of Fig. 2 (c) and (e).

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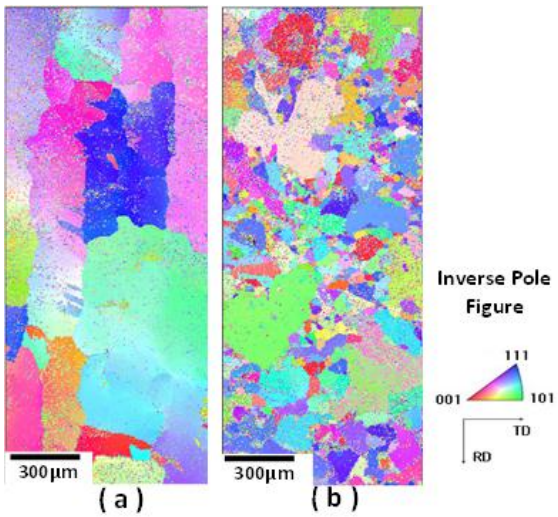


Fig. 3 EBSD mapping results of recrystallized texture of Fe-Co prepared by rolling for （a）1and （b）10 cycles

The mapping results of EBSD patterns of the recrystallized sample prepared by 1cycle and heating, and 10cycles and heating are shown in Fig. 3. The texture and crystal grain sizes in samples prepared by 1 and 10 cycles of cold-rolling were compared. The texture of the sample prepared by 1 cycle cold-rolling consisted of coarser grains. Microstructure and texture of 1 cycle consisted of distinguishing grains aligned along RD direction whose length and width are between few hundred microns to a millimeter and 100 and 500μm, respectively. On the other hand, the structure of the sample cold-rolled for 10 cycles underwent grain refinement and grains sizes varied between few microns and few hundred microns. Grains were randomly oriented with respect to RD direction [22]. To remove mechanical strain in samples that have been rolled or undergone strong processing, the samples were subsequently heat-treated above the phase transformation temperature. Consequently, the texture became randomly oriented and the detection of Kikuchi pattern was difficult. Thus, we measured the pole figures in the directions of {1 1 0}, {2 0 0}, {2 1 1}, {2 2 0}, {2 2 2}, {3 1 0} and constructed the ODF obtained from X-ray measurements to determine the orientation of the texture. EBSD ODF measurements of samples before and after annealing were considered quite essential. However, EBSD patterns were not clear due to severe deformation caused by cold rolling. Thus, we restricted the EBSD measurement on the annealed sample only as shown in Fig. 3.

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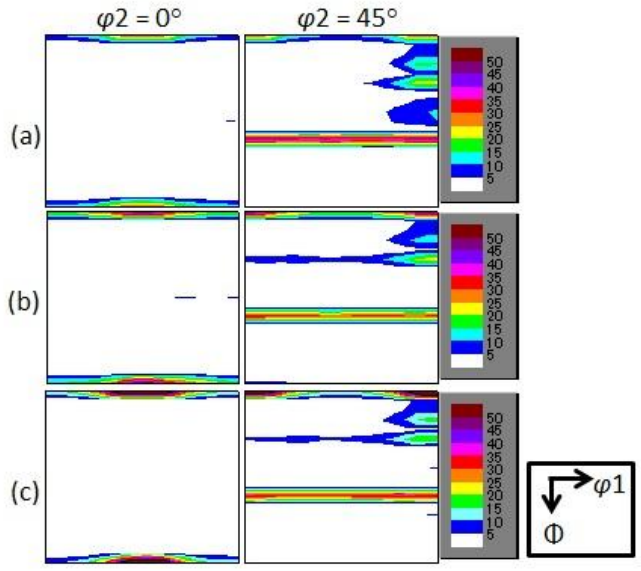


Fig. 4 ODF of (a) as-rolled (b) recrystallized and (c) nitrided (φ_2= 0, 45 deg.) samples after rolled for 1 cycle.

The ODF of (a) 1 cycle as-rolled, (b) recrystallized and (c) plasma-nitrided samples for φ_2 = 0, and 45 deg. are shown in Fig. 4. The appearance of rolled texture was specific to body centered cubic (BCC) structure. In 1 cycle as-rolled sample prepared aiming grain coarsening, maximum intensities were recorded for φ_2 = 0 deg., φ_1=45 deg. Φ= 0 and 90 deg. Similarly, maximum intensities were recorded for φ_2 = 45 deg. φ_1= 0 and 90 deg., Φ= 0 deg., which are characteristics of rotated-cube orientation in {100} <001>. However, the maximum intensity appeared at around φ_2=45 deg., Φ= 50 deg., was identified as γ- fiber. It is a typical characteristic of rolled textured BCC structured metals.

The ODF results of (a) as-rolled, (b) recrystallized and (c) nitrided textured samples prepared to aim grain refinement by rolling for 10 cycles are shown in Fig. 5. The maximum intensities were recorded at angles similar to 1cycle in as-rolled texture. In addition, the presence of γ-fiber was also identified. In the case of recrystallized texture too, the characteristics were similar to the results of 1cycle, but a considerable decrease in γ-fiber was observed.

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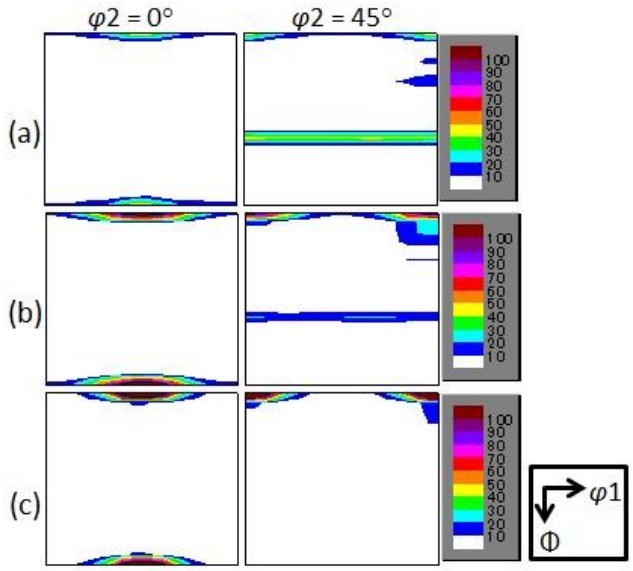


Fig. 5 ODF of (a) as-rolled (b) recrystallized and (c) nitrided (φ_2= 0, 45 deg.) samples after rolled for 10 cycles.

In the nitrided texture, γ-fiber was not identified at φ_2 = 45 deg. Rotated-cube orientation towards {100} <001> was more than the 1cycle case. We believe that the cube was rotated by 45 degrees to orient in {100} <001> in plasma nitrided texture [10][13][14][16].

When 1 and 10 cycle rolled textures were compared, the control of orientation was easy in 10 cycles textures than 1 cycle due to fine grains in the microstructure. Rotated-cube orientation was complete in nitrided texture and further study is necessary to understand the behavior of internal diffusion of nitrogen atoms during plasma nitriding in rolled texture.

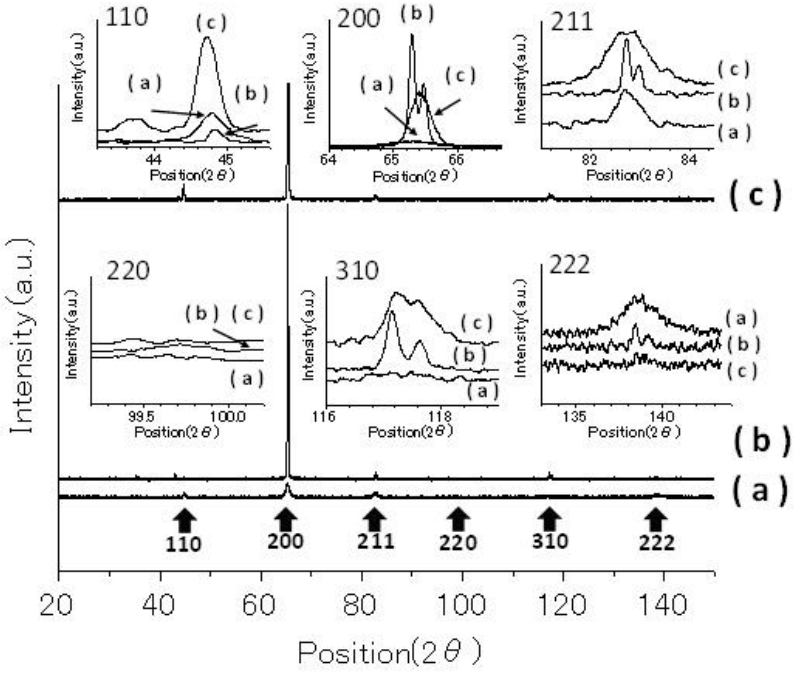


Fig. 6 XRD patterns of (a) as-rolled (b) recrystallized (650℃48h), and (c) Plasma nitrided (650℃ 48h) microstructures prepared by rolling for 10 cycles.

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Fig. 6 shows the results of XRD measurements of (a) as–rolled, (b) recrystallized and (c) nitrided microstructures.Six characteristic peaks 1 1 0, 2 0 0, 2 1 1, 2 2 0, 2 2 2 and 3 1 0 corresponding to BCC structure were identified in all three microstructures. The intensity of 2 0 0 peak was very significant. These characteristics expressed the possibility of {0 0 1} orientation in the normal direction (ND). The ratio of peak intensities between 1 1 0 and 2 0 0 for the above three samples were 1:3, 1:81, and 1:10, respectively. The ratio of recrystallized texture was markedly different from others and this is believed due to high crystallinity [20-21]. The microstructure and rotated-cube orientation in textures determined from ODF analysis and the XRD results agreed well. In the as-rolled sample, a single diffraction peak 200 appeared at 65.3 degrees and the peak width was wider than recrystallized and nitride samples. This indicated that the texture orientation of as-rolled sample was not complete and randomly orientated grains were very small in size. A split in the diffraction peak 200 for the recrystallized sample was recorded and peaked at 65.3 and 65.5 degrees. This is believed due to high crystallinity. On the other hand, a single peak in the vicinity of 65.4 degrees was observed for as–rolled, recrystallized samples. And also, the FWHM was greater than the recrystallized sample. This is considered due to the lattice distortion caused by nitrogen. However, to investigate the influence of nitrogen, the sample thickness of about 3 μm is not adequate enough. Thus, it is important to induce internal diffusion by introducing a third element that has higher affinity with nitrogen or considering nitridation by techniques other than plasma nitridation. This will increase the internal nitridation thickness and facilitate the analysis using various techniques.

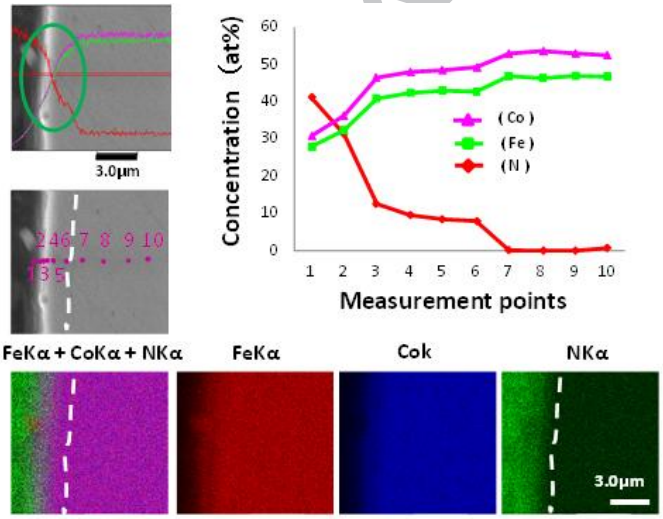


Fig. 7 The SEM image and elemental mapping (SEM-EDX) results of nitrided sample prepared by rolling for 10 cycles.

Fig.7 shows the SEM image and EDX results of plasma nitrided sample carried out to measure the diffusion behavior of nitrogen in Fe-Co alloy. Results confirmed that the preparation of homogeneous Fe-Co binary alloy with recrystallized texture by repeated cold-rolling and recrystallization using Fe and Co foils. Internal diffusion of nitrogen was observed up to 2.5 micrometers from the nitrided surface (white line in SEM image). This was confirmed by the line profile EDX analysis (red line). In contrast the diffusion of nitrogen atoms to the internal layer [23], nitrogen was able to diffuse inside of cold-rolled structures of Fe-Co alloy prepared by super- laminated layer method. We believe that this caused the distortion in the crystal structure, and consequently influenced the rotated cube orientation. Since the texture was controlled in the rotated-cube orientation, we expected the introduction of distortion in crystal structure by nitriding Fe-Co. Thus, the effect of rotated-cube orientation texture and interstitial nitrogen on magnetic properties of as-rolled, recrystallized, and nitrided texture samples were estimated.

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Magnetic hysteresis loops of (a) as-rolled, (b) recrystallized and (c) nitrided samples.by VSM measurements are also shown in Fig. 8. For comparison, magnetic measurements of Fe and Co foil, powder of crushed bulk prepared by arc melting and chemically synthesized Fe-Co particles

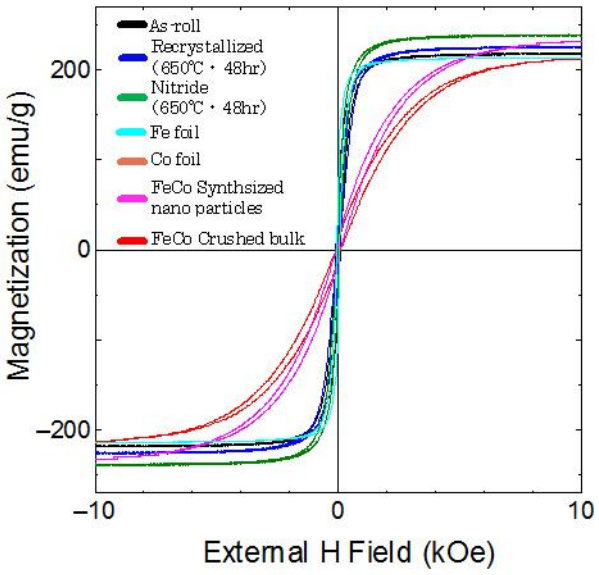


Fig. 8. Magnetic hysteresis loops of (a) as-rolled, (b) recrystallized and (c) nitrided samples.

The characteristics of the in-plane hysteresis loops of as-rolled, recrystallized and nitride Fe-Co samples were different from those of Fe and Co foils and Fe-Co powders. On the other hand, there was no considerable difference among the out-of-plane hysteresis loops of as-rolled, recrystallized and nitrided textures, except the coercivity of as-rolled texture was slightly higher than the rest. The magnetic properties of in-plane and out-of-plane properties of (a) as-rolled, (b) recrystallized and (c) nitrided samples are given in Table 1 and 2, respectively. As the Results of In-plane the saturation magnetization of nitrided texture was higher than other samples and assumed a value of 238.3 emu/g. The saturation magnetizations of as-rolled and recrystallized were 217.8 and 230.6 emu/g, respectively. The saturation magnetization of as-rolled sample was very close to the value of Fe foil.

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Table 1 Comparison of magnetic properties of in-plane of microstructure and texture dependence Fe-Co alloys prepared under different treatment conditions.

	As-rolled	Re-crystallized	Nitrided	Fe foil	Co foil	FeCo crushed bulk	FeCo particles
	In plane						
Magnetization Ms (emu/g)	217.8	230.6	238.3	226.2	159.5	211.6	231.3
Coercivity Hc (Oe)	87.7	46.3	40.9	42.0	55.1	87.8	69.2

Table 2 Comparison of magnetic properties of Out-of-plane of microstructure and texture dependence Fe-Co alloys prepared under different treatment conditions.

	as-rolled	re-crystallized	Nitrided	Fe foil	Co foil
	Out of plane				
Magnetization Ms (emu/g)	122.8	137.0	121.7	109.3	79.3
Coercivity Hc (Oe)	261.8	96.7	70.2	95.8	151.4

Table.1. shows the saturation magnetization and coercivity values of as-rolled, recrystallized and nitrided textures of Fe-Co sample prepared by rolling for 10 cycles. For comparison, the in-plane magnetic measurements of Fe and Co foil. Table.2. shows the out-of- plane magnetic properties.

Though there was a slight difference in the coercivity value, crystal structure distortion is not large enough to bring about a dramatic change in the overall magnetic properties. Considering the structural changes observed in as-rolled, recrystallized and nitrided Fe-Co samples, we believe that the distortion along the C axis could be realized by using carbon as an interstitial element along with other elements such as Al, which has a high affinity for nitrogen.

3. Conclusions

In this study, we established a technique to prepare microstructures and textures of Fe-Co alloy by combining repeated cold-rolling and recrystallization. This has made the alignment controlled texture to rotated-cube orientation, which is the typical orientation of magnetic easy axis, and crystal grain size refinement possible. Consequently, the magnetic properties of controlled texture Fe-Co alloy were different from Fe-Co alloy powder and foil. Though the internal diffusion of nitrogen was detected in plasma nitrided texture of Fe-Co alloys, the crystal structure distortion was not large enough to bring about a remarkable difference in the magnetic properties. We believe that the magnetic properties of Fe-Co can be by bringing about considerable distortion by introducing carbon as interstitial element along with the addition of other elements such as Al, and V that has high affinity for nitrogen.

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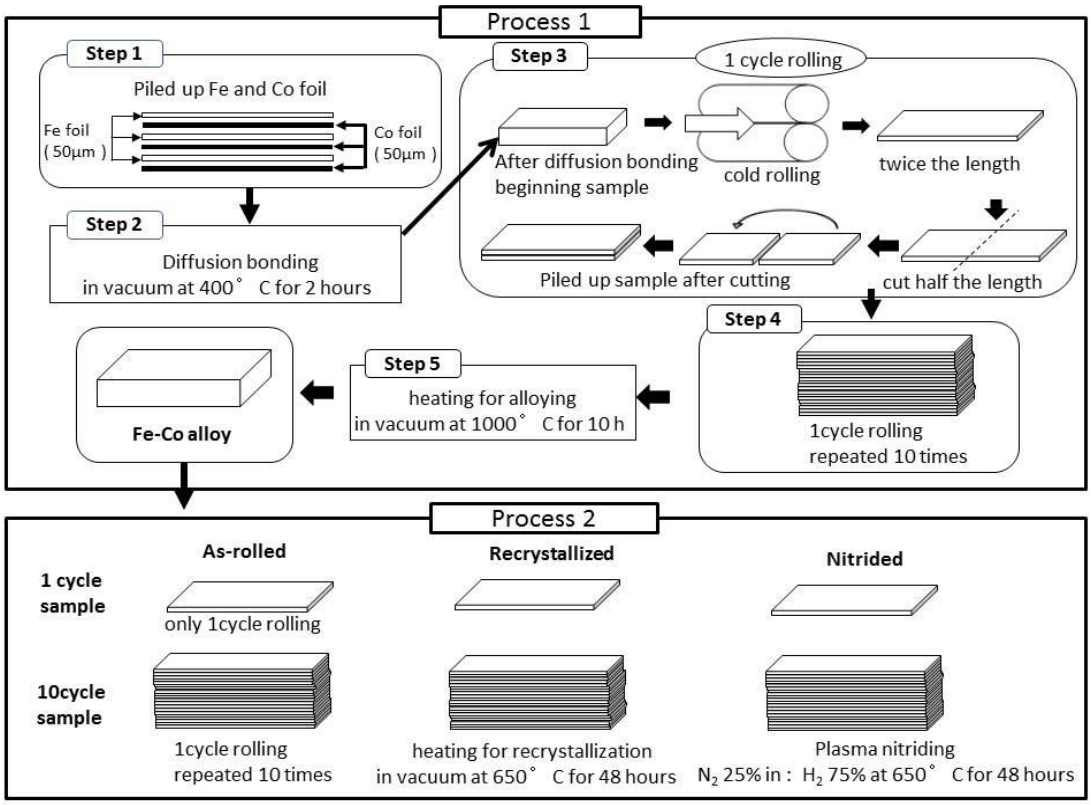


Fig . 1 . Procedure and preparation of stallization and nitride sample

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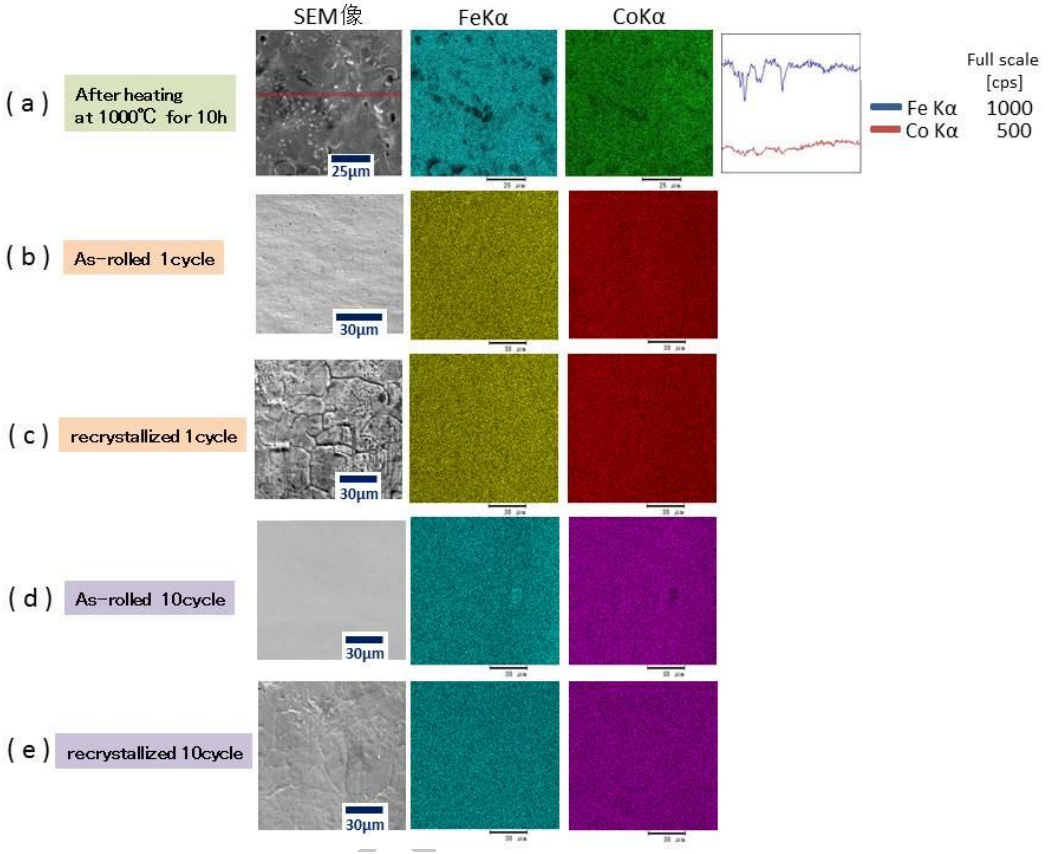


Fig. 2. SEM-EDX (a) after heating at 1000℃for 10h (b) As-rolled 1 cycle (c) recrystallized (d) As-rolled 10 cycles and (e) recrystallized 10cycles samples .

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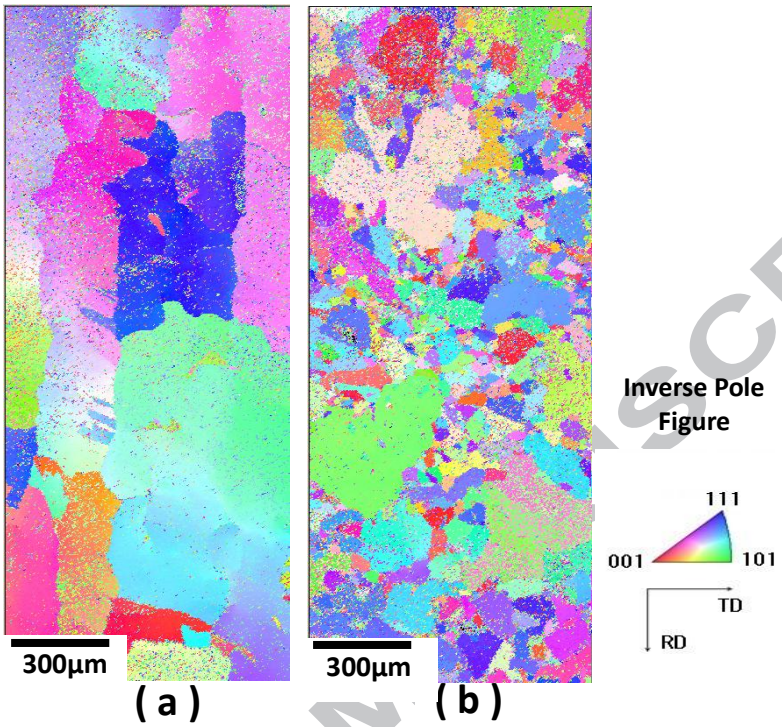


Fig. 3 EBSP mapping results of recrystallized texture of Fe-Co prepared by rolling for (a) 1and (b) 10 cycles

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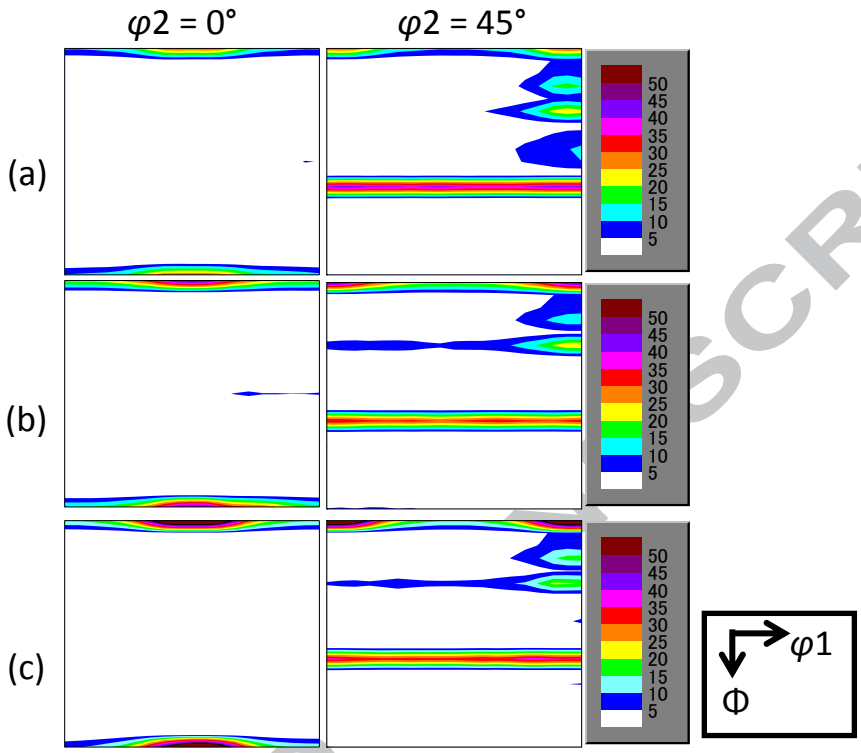


Fig.4 ODF after 1 cycle rolling texture(a) As-rolled (b) Recrystallized and (c) Nitride (φ_2= 0, 45 deg.)

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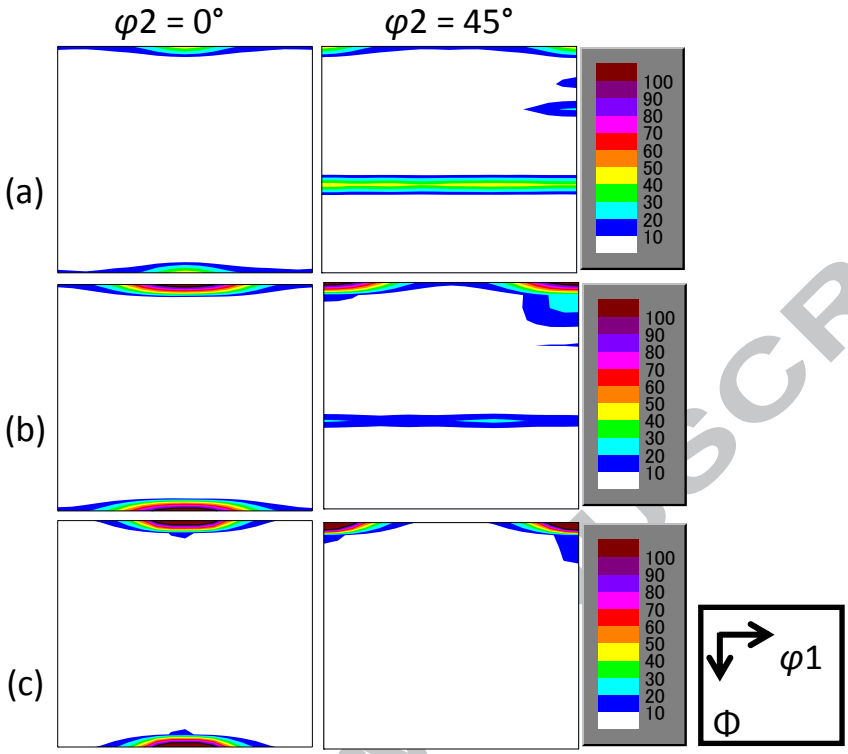


Fig. 5 ODF of (a) as-rolled (b) recrystallized and (c) nitrided (φ_2= 0, 45 deg.) samples after rolled for 10 cycles.

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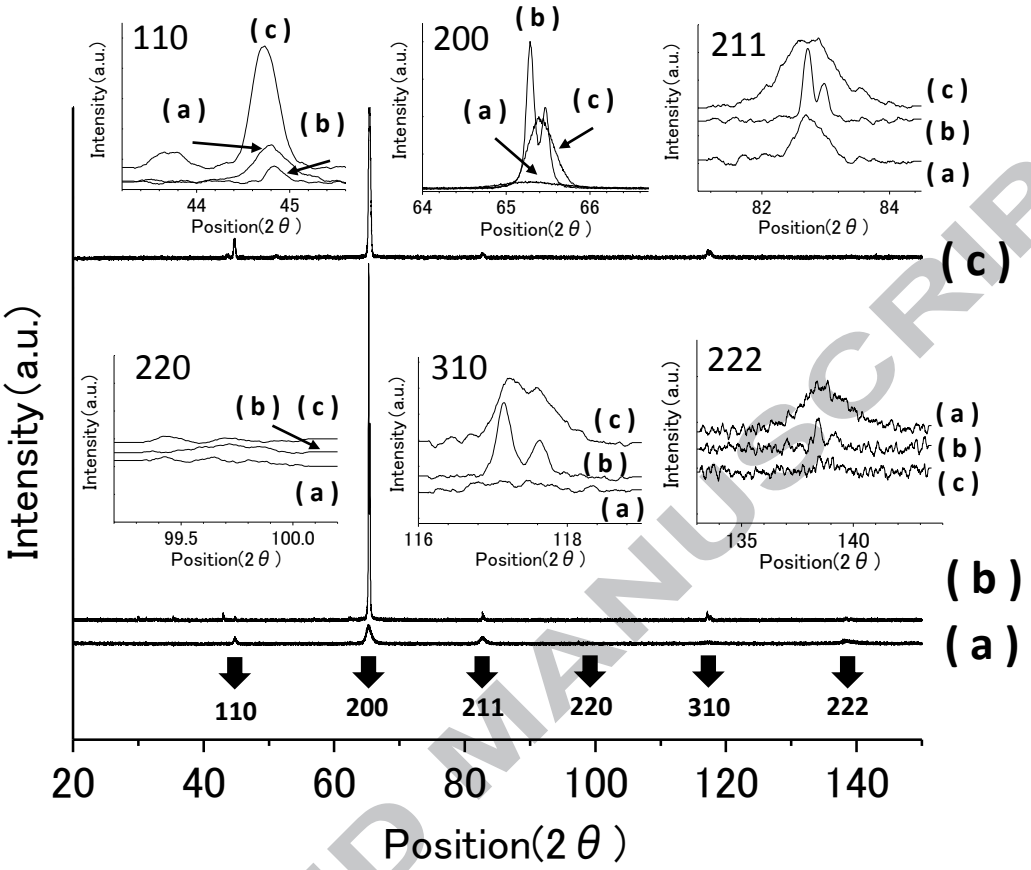


Fig. 6 XRD patterns of (a) as-rolled (b) recrystallized (650℃48h), and (c) Plasma nitrided (650℃48h) microstructures prepared by rolling for 10 cycles.

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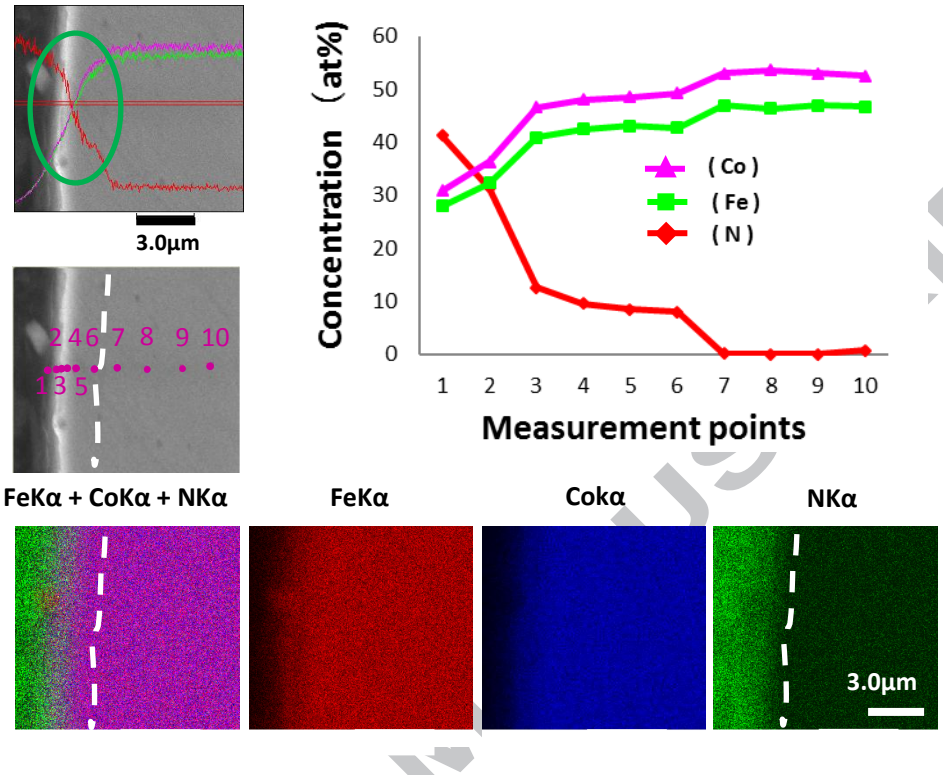


Fig. 7 The SEM image and elemental mapping (SEM-EDX) results of nitrided sample prepared by rolling for 10 cycles.

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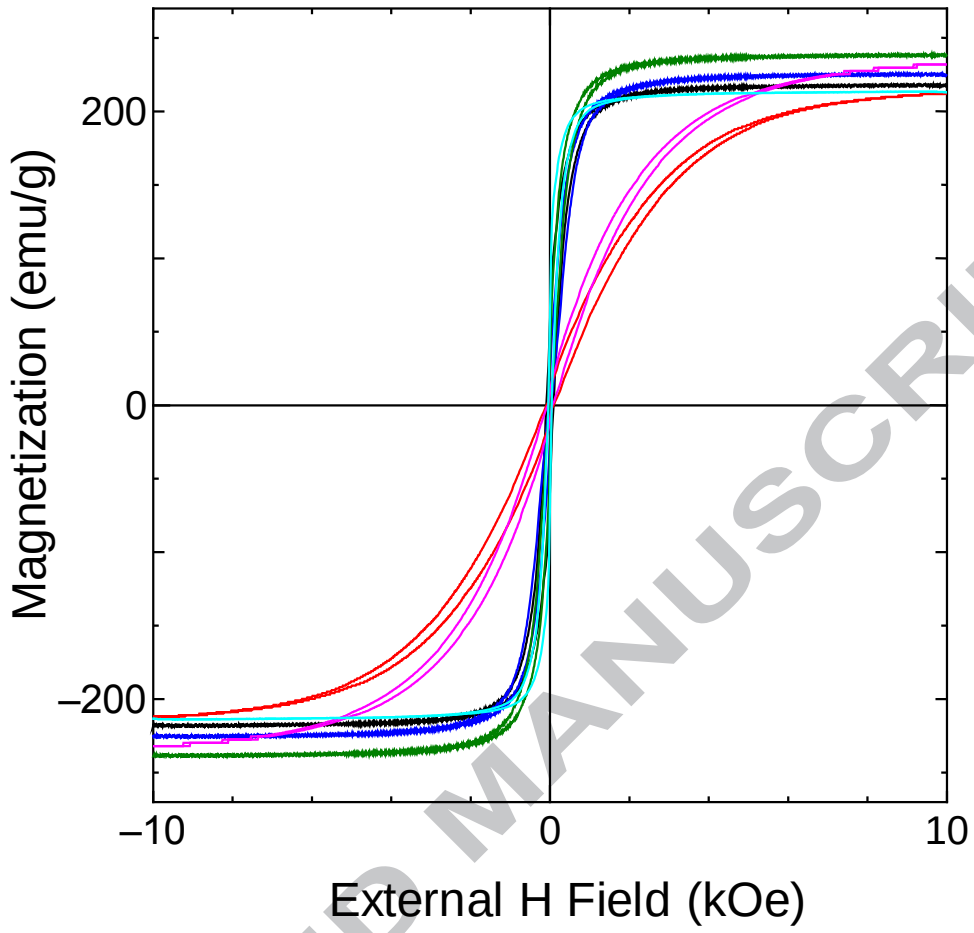


Fig. 8 Magnetic hysteresis loops of (a) as-rolled, (b) recrystallized and (c) nitrided samples.

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Table 1 Comparison of magnetic properties of microstructure and texture dependence Fe-Co alloys prepared under different treatment conditions.

	As-rolled	Re-crystallized	Nitrided	Fe foil	Co foil	FeCo crushed bulk	FeCo particles
	In plane						
Magnetization Ms (emu/g)	217.8	230.6	238.3	226.2	159.5	211.6	231.3
Coercivity Hc (Oe)	87.7	46.3	40.9	42.0	55.1	87.8	69.2

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Table 2 Comparison of magnetic properties of Out-of-plane of microstructure and texture dependence Fe-Co alloys prepared under different treatment conditions.

	as-rolled	re-crystallized	Nitrided	Fe foil	Co foil
	Out of plane				
Magnetization Ms (emu/g)	122.8	137.0	121.7	109.3	79.3
Coercivity Hc (Oe)	261.8	96.7	70.2	95.8	151.4

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Highlights

· Preparation of microstructure and texture controlled Fe-Co alloy by repeated cold rolling and recrystallization using Fe and Co foils as starting material was achieved successfully.

· Crystal grain sizes of microstructure and alignment of the textures in the direction of easy axis of magnetization of rotated-cube were confirmed by EBSD and ODF measurements.

· Internal nitriding of Fe-Co alloy by plasma nitriding was confirmed and facilitated texture orientation.

· Magnetic properties of as-rolled, recrystallized and nitrided Fe-Co were measured found similar to bulk Fe-Co alloy

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