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**BIAXIALLY TEXTURED COPPER
AND COPPER-IRON ALLOY
SUBSTRATES FOR USE IN
 $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ COATED CONDUCTORS**



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14. ABSTRACT Copper and Cu-Fe (Fe ~ 2.35 wt%) alloy substrates were thermo-mechanically processed and the biaxial texture development, magnetic properties, yield strength, and electrical resistivity were studied and compared to determine their suitability as substrates for high-temperature superconducting coated conductor applications. Average full width half maximum (FWHM) of 5.5° in Phi scans (in-plane alignment), and 6.6° in omega scans (out-of-plane alignment) was obtained in copper samples. Cu-Fe samples showed 5.9° FWHM in Phi scans and 5.9° in omega scans. Even with the presence of 2.35% Fe in the Cu-alloy, the saturation magnetization (M_{sat}) value was found to be 4.27 emu g ⁻¹ at 5 K, which is less than in Ni samples by an order of magnitude and comparable to that of Ni-9 at.% W substrates. The yield strength of the annealed Cu-Fe alloy substrate was found to be at least two times higher than that of similarly annealed copper substrates. The electrical resistivity of Cu-Fe alloy was found to be an order of magnitude higher than that of pure copper at 77 K.					
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Biaxially textured copper and copper–iron alloy substrates for use in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ coated conductors

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Abstract

Copper and Cu–Fe (Fe $\sim$ 2.35 wt%) alloy substrates were thermo-mechanically processed and the biaxial texture development, magnetic properties, yield strength, and electrical resistivity were studied and compared to determine their suitability as substrates for high-temperature superconducting coated conductor applications. Average full width half maximum (FWHM) of 5.5° in Phi scans (in-plane alignment), and 6.6° in omega scans (out-of-plane alignment) was obtained in copper samples. Cu–Fe samples showed 5.9° FWHM in Phi scans and 5.9° in omega scans. Even with the presence of 2.35% Fe in the Cu-alloy, the saturation magnetization (M_{sat}) value was found to be 4.27 emu g^{-1} at 5 K, which is less than in Ni samples by an order of magnitude and comparable to that of Ni–9 at.% W substrates. The yield strength of the annealed Cu–Fe alloy substrate was found to be at least two times higher than that of similarly annealed copper substrates. The electrical resistivity of Cu–Fe alloy was found to be an order of magnitude higher than that of pure copper at 77 K.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Recent advancements in high-temperature superconducting (HTS) coated conductor technology have resulted in up to 30 m long second-generation $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) tapes that can carry high currents being able to be fabricated on textured metallic substrates with suitable buffer layers [1]. These advancements have been made primarily using Ni or Ni-alloy substrates, especially Ni–W [2–4]. An attractive alternative to Ni-based substrates may be Cu-based substrates since copper is six times cheaper than nickel on a kilogram per kilogram basis. The lattice parameter for crystalline Cu is also sufficiently close to that of the YBCO, making it a potential candidate. Past work has demonstrated the potential for copper to be textured, although the texture has been sub-par compared to Ni [5]. Only recently has copper been demonstrated with good $\{100\}$ $\{100\}$ texture [6, 7].

Another advantage of copper over nickel is its low resistivity. YBCO-coated conductors have an additional Cu

layer placed on top of the Ag protective layer to serve as a stabilizing layer. The stabilizing layer serves two purposes, offering a current path in the event of a local quench in the coated conductor as well as having high thermal conductivity to dissipate heat from the quench location and bring the conductor back into the superconducting state. However, this layer is generally thick, being tens of microns in thickness, which can significantly lower the engineering current density (J_E) of the HTS-coated conductor. If the YBCO layer can be electrically (and potentially thermally) connected to a copper substrate, it is possible to eliminate the Cu stabilizing layer and therefore increase J_E .

To make the copper substrate practical, alloying will be necessary to improve the mechanical durability of the tape. This is one of the reasons for using a Ni-alloy as opposed to pure Ni. However, alloying will increase the resistivity of the Cu and, as such, dispersion strengthening of the copper may provide an alternative to improve the mechanical strength while maintaining adequate conductivity. In this sense, an improved

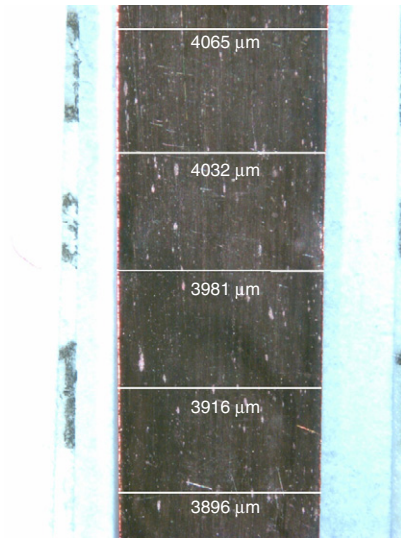


Figure 1. Image of width measurements on a mounted biaxially textured Cu substrate specimen onto a cardboard holder with 22 mm long tensile gauge section cutout.

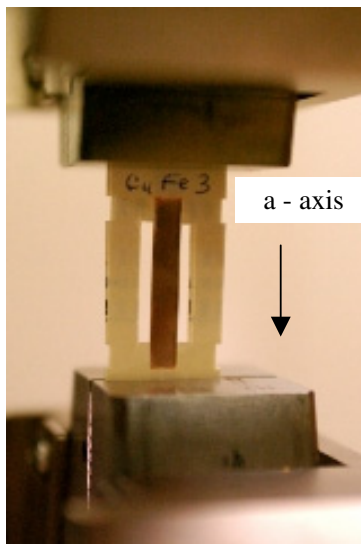


Figure 2. Photograph of a biaxially textured specimen Cu-Fe3 (third sample of Cu-Fe alloy) that is aligned and mounted into a tensile grip. The rolling direction and *a*-axis are along the length of the sample.

dc HTS-coated conductor may be realized through the copper substrates.

However, in ac applications such as generators and motors other considerations must be made [8]. The ac losses resulting from eddy currents in the highly conductive copper will become important and may prevent its use in these ac applications [9, 10]. Ferromagnetic losses [11, 12] may also be introduced depending on how the copper, which is nonmagnetic, is alloyed. As such, a significant distinction must be made between copper-based substrates for a dc conductor and that attempted for an ac conductor. The Cu-alloy for an ac conductor must be made with a high resistivity as opposed to the low resistivity for the dc conductor. The advantage of a substrate-stabilized conductor previously mentioned is then

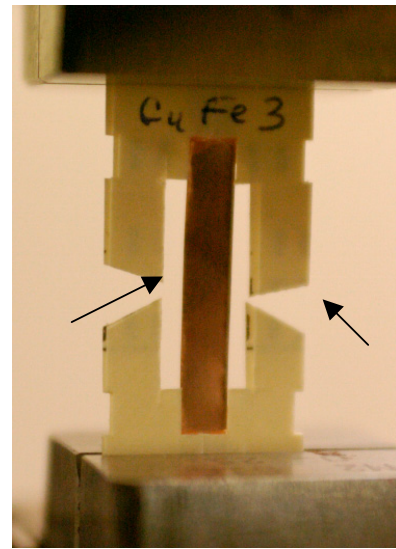


Figure 3. Photograph of the biaxially textured Cu-Fe specimen (Cu-Fe3) after the cardboard used for alignment was cut (before testing).

nullified, with the primary advantage of copper over Ni being the cost per kilogram if being used for an ac conductor. The use of copper-based substrates will therefore be more applicable to a dc conductor. Copper-based alloys such as Cu-Ni [13, 14], Cu-Ni-Mn [13] and Cu-Ni-Al [7] have been investigated for developing the biaxial texture.

Electrical and magnetic properties of Cu-Fe alloys with low Fe amounts (<100 ppm) were studied by Fickett [15]. However, the texture development in these alloys was not studied earlier. In the present study, a Cu-Fe alloy with higher amounts of Fe additions (~2.35 wt%) was processed to obtain highly textured substrates with good strength as alloying generally increases the yield strength. In addition to the electrical and magnetic properties, the texture development, yield strength, etc, of these Cu-Fe alloy substrates were also studied and compared with pure copper.

2. Experimental details

High-purity copper (99.99%) and a Cu-Fe (Fe 2.35 wt%, P 0.03 wt%, Zn 0.12 wt%, ASTM spec. No B465) alloys were used in this study. X-ray fluorescence (XRF) analysis was used to confirm the composition. Copper rods of starting dimension 9.5 mm diameter were pre-annealed at 450 °C for 1 h and then reverse cold rolled to get a 99.5% total reduction in thickness using a 10% reduction per pass schedule. The final thickness of the copper samples was around 30–40 μm. Highly polished rolls were used to get a smooth surface finish in the samples. The copper samples were annealed in a high-temperature vacuum box furnace at 750 °C in an Ar/H₂ atmosphere for 1 h before cooling to room temperature. Results obtained on copper samples processed in a tube furnace are given elsewhere [6]. The Cu-Fe sheet samples were rolled at the manufacturer facility (Olin Corporation) to a desired thickness of ~50 μm. These samples were vacuum sealed in quartz ampoules and then annealed in a box furnace at 1000 °C for 1 h. For the Cu-Fe samples used in the mechanical

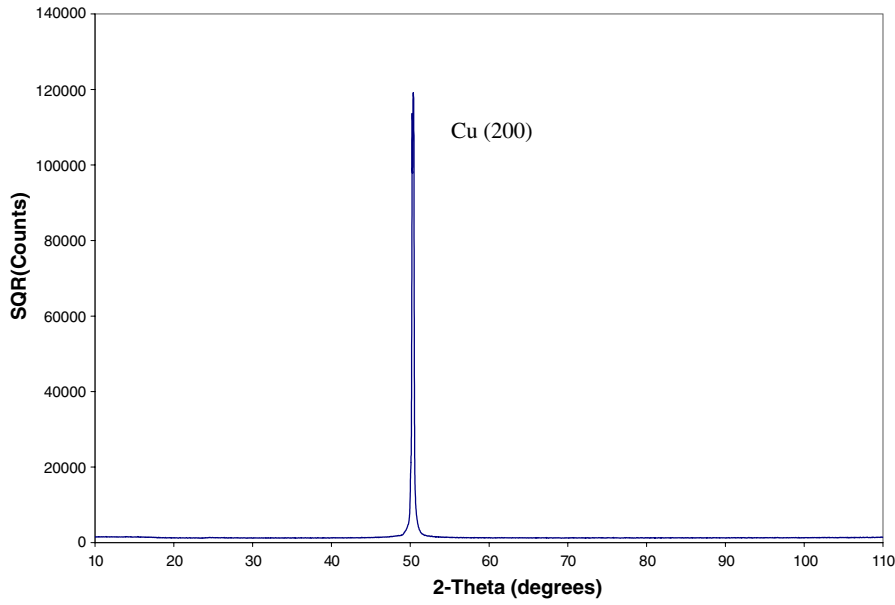


Figure 4. An x-ray theta-two-theta scan of a textured Cu sample showing essentially *c*-axis texture.

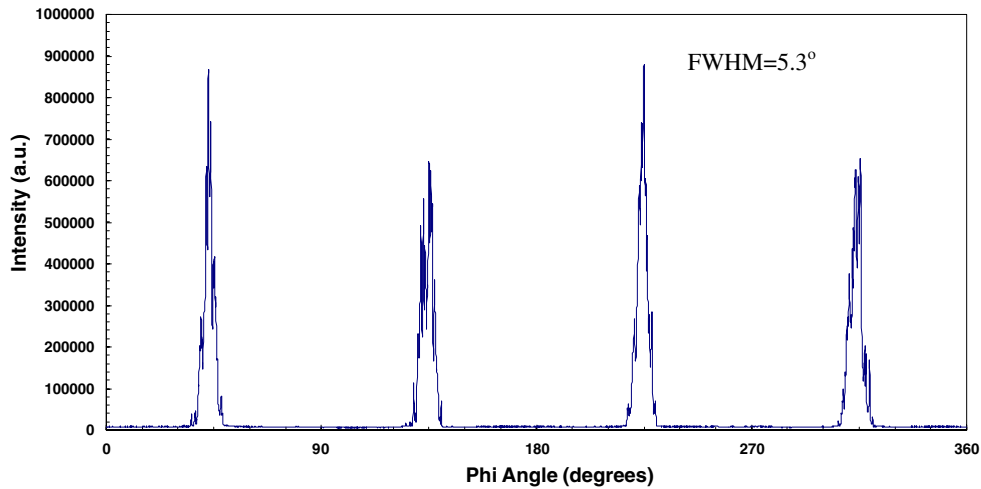


Figure 5. An x-ray (111) phi scan of a Cu sample. An average FWHM of 5.3° is obtained in this sample.

properties test, annealing was also done in a tube furnace along with copper substrates at 750°C for 1 h.

The texture present in these samples was determined by using two-theta, phi, and omega scans and x-ray pole figures using a Philips x-ray diffractometer. Since the samples have large grains ($\sim 100\ \mu\text{m}$), the roughness or scatter in the data is possible due to the individual points that come from these grains. The data were collected with a 0.1° step for phi scans and 0.01° step for omega scans. The full width half maxima (FWHMs) of the phi and omega scans were determined from a Gaussian curve fitted to the data to eliminate the errors in the FWHM measurement. An average FWHM value of four peaks in phi scans is used to represent the FWHM of that particular sample.

Orientation imaging microscopy (OIM) was used to obtain the grain misorientation maps on the samples at several points roughly over a $500\ \mu\text{m} \times 800\ \mu\text{m}$ area. Magnetization data

at 5 and 77 K for the Cu-Fe samples were collected by using a vibrating sample magnetometer (Quantum Design PPMS) both before and after the annealing treatments. The magnetic field in the magnetometer was applied parallel to the sample to reduce the demagnetization effects. The electrical resistivity was measured by using a standard four-probe method at both 77 K and at room temperature.

In order to keep the heat treatment history the same, both copper and Cu-Fe samples were annealed at 750°C in a tube furnace for the specimens used in the mechanical property measurement. The texture analysis was done on both Cu and Cu-Fe samples annealed at 750°C prior to mechanical treatment to verify that cube texture is present in all the samples. Both copper and Cu-Fe samples were annealed at 450°C for 1 h after cutting the foils to the desired size, to reduce the work-hardening effects at the cutting edges of the samples prior to the tensile testing.

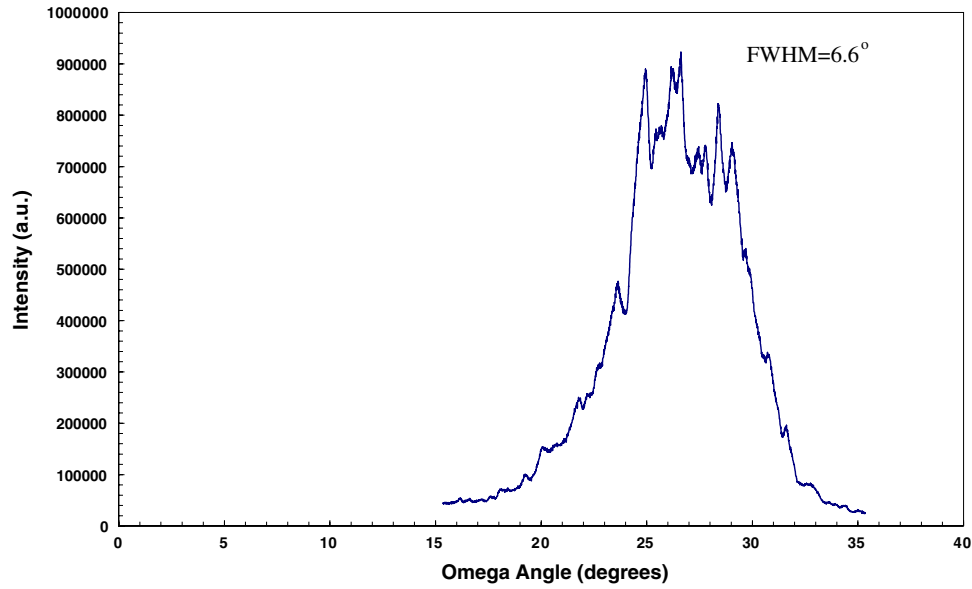


Figure 6. An x-ray (200) omega scan taken in the parallel to rolling direction of a Cu sample with an FWHM of 6.6°.

Table 1. The FWHM of phi scans for different Cu samples taken from a long tape. The values noted in the table reflect the average values of the FWHM values of the four peaks in the associated phi scan.

Sample	Phi (deg)
1	6.4
2	5.5
3	5.2
4	5.7
5	4.7
Average	5.5

The yield strengths of annealed copper and Cu-Fe foil substrates were determined by using an Instron tensile testing machine. A similar procedure was followed for the sample preparation and the testing for all three sets of samples. The purpose of the tensile test is to compare the yield strengths of similarly processed pure copper and copper alloy textured substrates. Since the coated conductors that are being developed for HTS applications will use metallic substrates that have a $\sim 3\text{--}4$ mm width and a small thickness of $\sim 50\text{ }\mu\text{m}$ (for higher J_E), foil samples that were 3 mm wide and 0.05 mm ($50\text{ }\mu\text{m}$) thick were selected for the tensile testing. The samples were cut such that the long axis of the sample is parallel to the a -axis (100) (rolling direction). Since the specimen size is atypical, ASTM D3379 (standard test method for tensile testing of single filament materials) was used as a guide to test the tensile strength of foils. Each foil specimen was aligned and epoxied to a piece of cardboard with a 22 mm long gauge section cut out as per ASTM D3379. The specimen width was determined from a minimum of five measurements along the tensile gauge section length. Figure 1 shows a typical width measurement taken from one of the foil samples. The thickness of the samples was measured prior to mounting on the cardboard. The cardboard and foil specimen were aligned and mounted into the tensile wedge grip as shown in figure 2. Before testing, the cardboard was cut with scissors

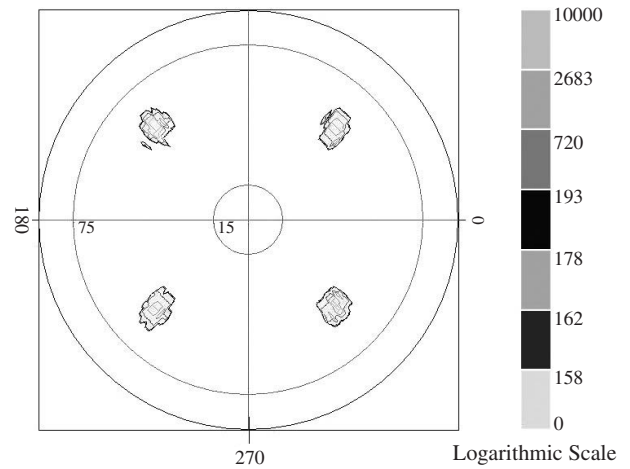


Figure 7. (111) x-ray pole figures in log scale for a Cu sample annealed at 750 °C for 1 h.

as shown in figure 3 so that when the load is applied it will be carried only by the sample. The crosshead rate was at 0.508 mm min^{-1} (0.02 in min^{-1}) and no extensometer or strain gauges were used. From the load versus displacement data from the Instron testing machine, pseudo stress versus strain plots were generated.

3. Results and discussion

Figure 4 shows the theta-two-theta scan of a rolled and annealed copper sample showing essentially the (200) reflection of pure copper. Figure 5 shows the (111) Phi scans taken from a sample cut from a long copper tape. Table 1 shows the four-circle x-ray diffraction data obtained from five different samples cut randomly but not sequentially from the copper tape sections. It can be seen that the average FWHM

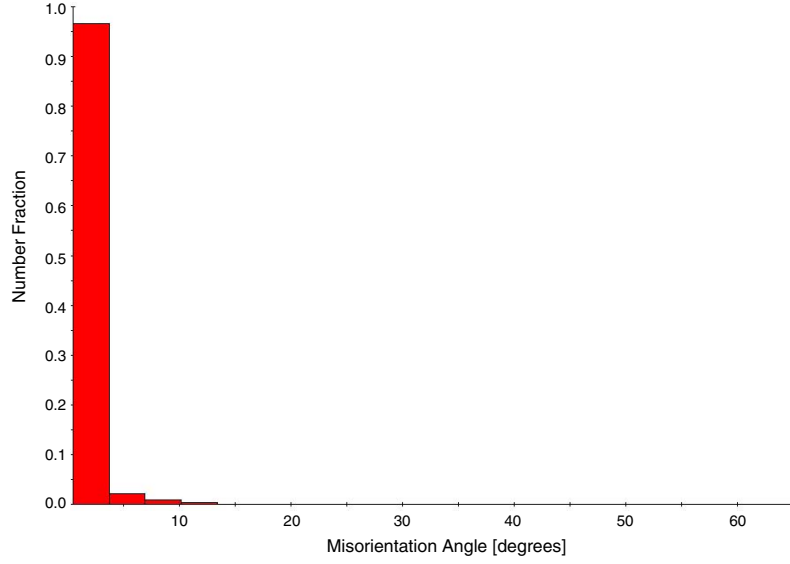


Figure 8. The grain misorientation angle distribution as determined by OIM for a textured Cu sample.

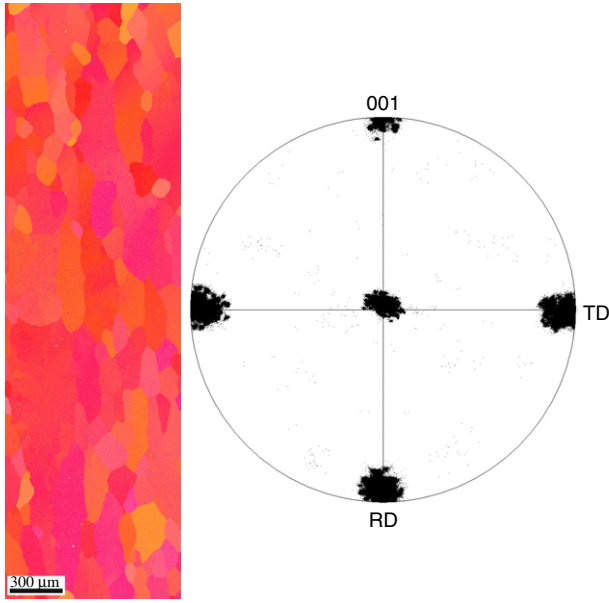


Figure 9. The (001) OIM pole figure and the area of the sample studied in a textured copper substrate.

of the phi scan peaks (in plane grain alignment) is $\sim 5.5^\circ$. Although some sections of the samples showed an FWHM of 4.7° , the range varied from 4.7° to 6.4° . Figure 6 shows the omega scans taken in the rolling directions of one of the samples. The FWHMs of the omega scans were found to be 6.6° , showing good out-of-plane alignment. Figure 7 shows the x-ray (111) pole figures in log scale, showing essentially reflections from the (100) planes.

Figure 8 shows the orientation image microscopy (OIM) data taken from one of the Cu samples. It reveals that the samples predominantly have low-angle grain boundaries. These data corroborate with the findings of a high degree of texture as evidenced in the x-ray diffraction scans given

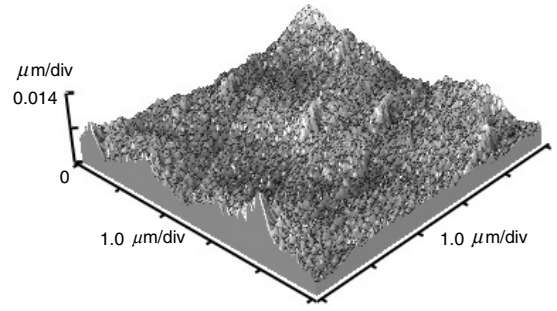


Figure 10. An atomic force micrograph for the (001) textured Cu sample. The surface roughness (rms) is about 2.7 nm.

Table 2. Magnetization data obtained at 5 K for different metallic substrates.

Material	M_{sat} (emu g $^{-1}$)
Nickel ^a	57.06
Ni–3 at.% W ^a	36.4–37.3
Ni–9 at.% W ^a	4.36
Cu–Fe (this study)	4.27

^a From [11].

Table 3. Electrical resistivity data obtained at 77 and 300 K (room temperature) for annealed Cu and Cu–Fe substrates used in this study.

Temp (K)	Annealed Cu–Fe (Ω m)	Annealed Cu (Ω m)
300	3.8×10^{-8}	1.6×10^{-8}
77	2.44×10^{-8}	1.85×10^{-9}

previously. Figure 9 shows the OIM pole figure using the raw data for a copper sample along with the area of the sample that is analysed depicting the (001) texture and absence of any other reflections from the samples. The data given here indicate an

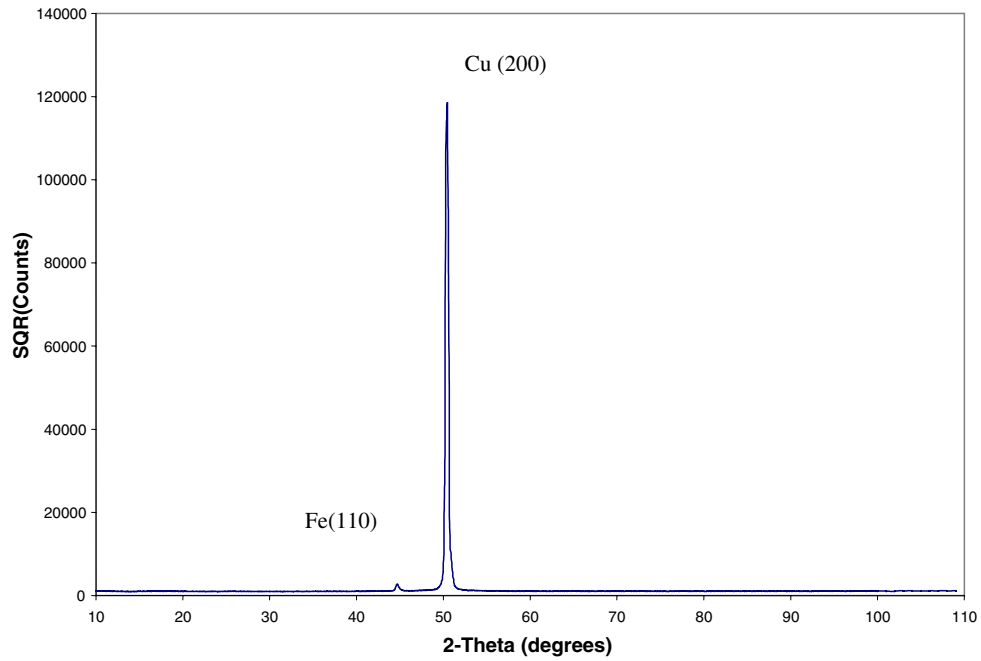


Figure 11. X-ray theta–two-theta scans of a Cu–Fe sample showing high c -axis texture and a peak from Fe precipitates.

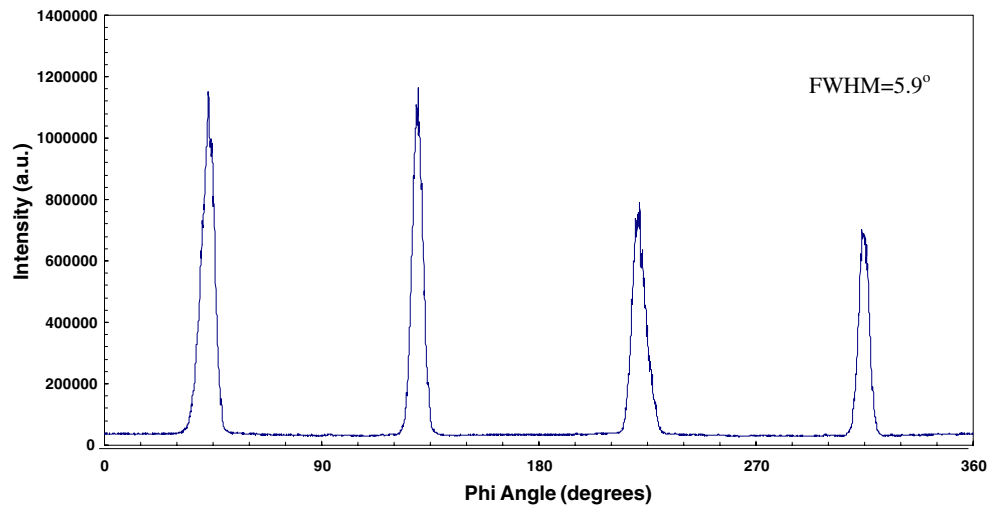


Figure 12. A (111) phi scan of a Cu–Fe sample treated at 1000 °C. The average FWHM is 5.9°.

overall improvement in the grain alignment of the copper from a previously published report [6].

Figure 10 shows an atomic force microscope (AFM) picture taken from the surface of a pure Cu sample. A surface roughness of 2.7 nm (rms) was measured in this particular sample. Generally the surface roughness was found to vary from 2–8 nm (rms) when measured on several samples. The rolls used in the present experiment were polished to 1 μ m. The final surface finish of the copper samples directly depends upon the surface finish of the rolls [6]. The surface of the textured copper substrates was also noted to get smoother after the annealing treatment.

Since the Cu–Fe samples were not rolled using the polished rolls, the surface was found to be rougher than pure

copper substrates, as expected. However, after the annealing treatment, the processed Cu–Fe substrates also displayed good texture. Figure 11 shows the theta–two-theta scan for a Cu–Fe sample showing high c -axis texture. The second peak observed at two-theta of 44.7° corresponds to Fe(110) and is from the precipitates that were observed in the microstructure as discussed later. Figure 12 shows the phi scans of Cu–Fe samples processed at 1000 °C. An average FWHM of 5.9° was obtained in this sample. This value represents good in-plane alignment of the grains and is suitable for YBCO-coated conductor applications. As shown in figure 13, an FWHM of 5.9° was obtained for the omega scans, indicating that the out-of-plane alignment of the grains is also within the range of suitability for the YBCO-coated conductor. Figure 14 shows

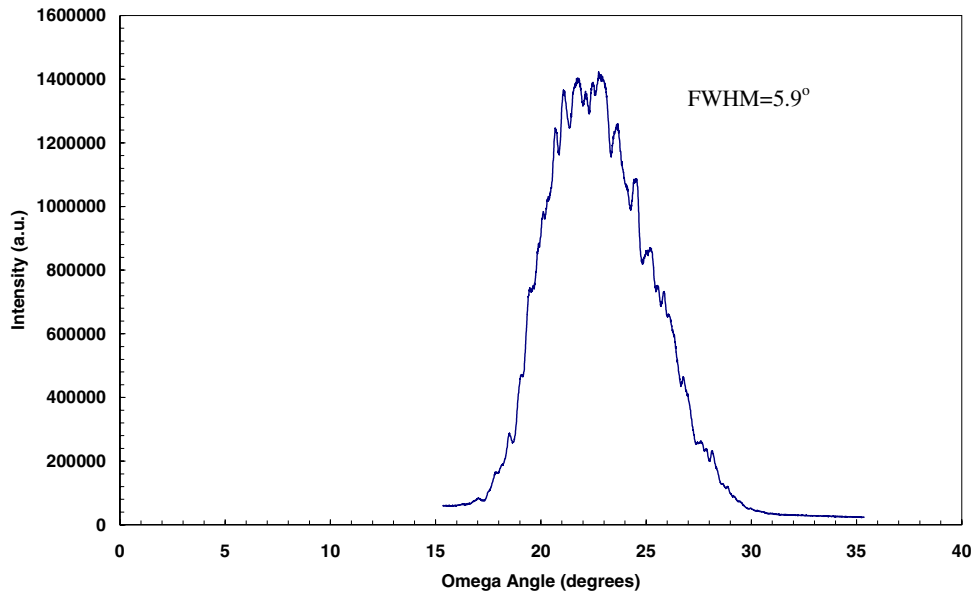


Figure 13. A (200) omega scan of a Cu-Fe sample annealed at 1000 °C. The FWHM is 5.9°.

the (111) x-ray pole figures in log scale for a Cu-Fe sample showing a high degree of cube texture.

Figure 15 shows the representative orientation image microscopy (OIM) data of a Cu-Fe sample. The data reveal that these samples also have grains with predominantly low-angle grain boundaries. Figure 16 shows the (001) raw data pole figure along with the area of the sample that is considered for the analysis from this sample, indicating again good quality texture for these samples as evidenced by the absence of other reflections.

Figure 17 shows the magnetization data taken from an as-rolled Cu-Fe sample at 5 K. It can be seen that the saturation magnetization (M_{sat}) is 4.27 emu g^{-1} . A slight reduction in the M_{sat} value was observed in the samples after annealing. By comparison with the published data in the literature as shown in table 2, the Cu-Fe sample has very low magnetization and is comparable to Ni-W (9 at.%), indicating that even with the presence of Fe in these alloys, the magnetic contribution is very small due to the small amount of Fe. There was no significant difference observed between 5 and 77 K measurements.

Table 3 shows the electrical resistivity data of both Cu and Cu-Fe textured substrates measured at 77 K and at 300 K (room temperature). It can be seen that resistivity of the Cu-Fe sample is comparable to pure Cu at room temperature. As the temperature is lowered, both the samples show a decrease in the resistivity as expected for metals and alloys. However, when the resistivity values of these two samples are compared at 77 K, it can be seen that the resistivity of Cu-Fe sample is higher than that of pure Cu by almost an order of magnitude. Higher resistivity of Cu-Fe sample at 77 K as compared to pure Copper may be viewed as a beneficial feature for this alloy, as an ac conductor based on these substrates may have reduced eddy current losses. The observed increase in resistivity with Fe additions to Cu is consistent with earlier findings of Fickett [15].

Since the mechanical properties were measured on the Cu and Cu-Fe substrates annealed at 750 °C, the presence

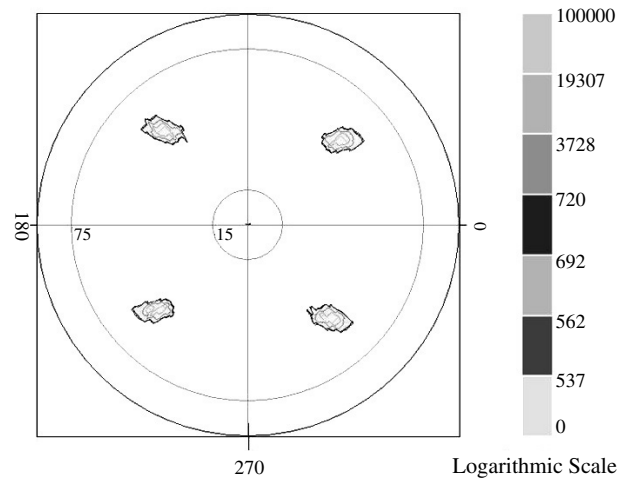


Figure 14. (111) x-ray pole figure in log scale of Cu-Fe sample annealed at 1000 °C, 1 h.

of cubic texture in these samples needs to be verified in the Cu-Fe samples. Figure 18 shows the phi scan taken from a Cu-Fe sample that was processed at 750 °C in a tube furnace. It can be seen that these substrates also have high degree of cube texture ($\text{FWHM} = 6.4^\circ$). The low amounts of non-cubic components (<3%) noticed in the substrates treated at 750 °C were expected not to influence the tensile strength considerably. The high-temperature anneal at 1000 °C has resulted in obtaining samples without non-cubic components (~100% cube texture) and better FWHM, as shown in figure 12. The omega scans on the substrates annealed at 750 °C also showed an FWHM of 6.4° parallel to the rolling direction, again indicating a good out-of-plane alignment of the grains.

From the load versus displacement data acquired from the Instron testing machine, pseudo stress versus strain plots were

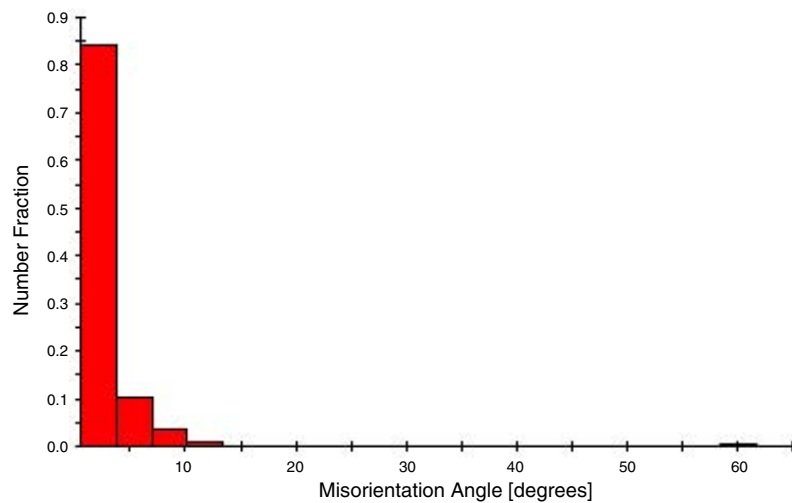


Figure 15. The grain misorientation angle distribution determined by OIM in a textured Cu–Fe sample treated at 1000 °C.

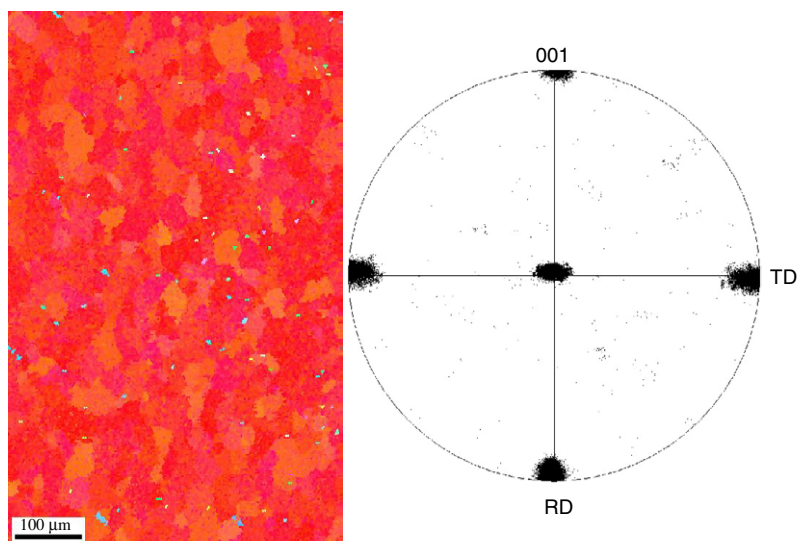


Figure 16. The (001) OIM pole figure and the area of the sample studied in a Cu–Fe sample.

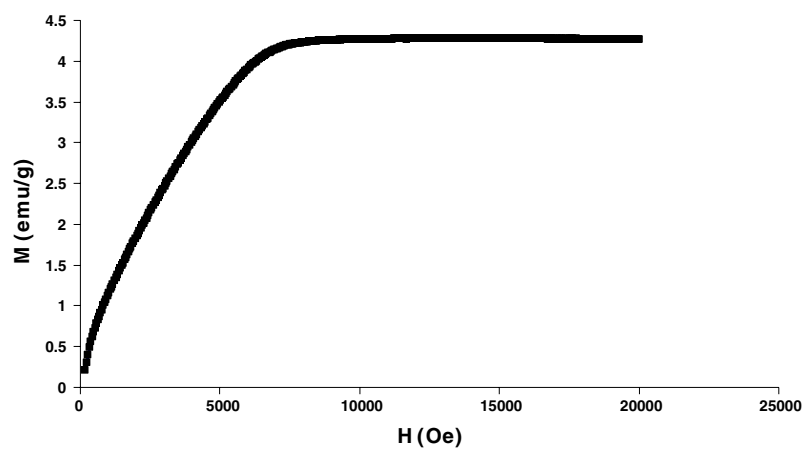


Figure 17. Magnetization data of Cu–Fe sample taken at 5 K. M_{sat} is 4.27 emu g^{−1}.

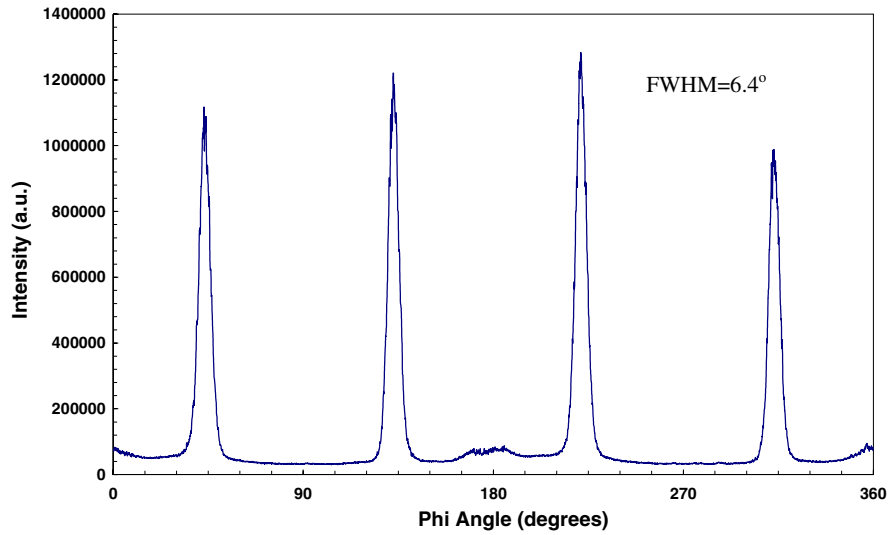


Figure 18. A (111) phi scan of a Cu–Fe sample treated at 750°C. The average FWHM is 6.4°. These samples were used for the yield strength determination. Figure 12 shows the phi scan of a Cu–Fe sample treated at 1000°C.

Table 4. Tensile test data taken from three sets of Cu and Cu–Fe samples.

ID	Average width (mm)	Thickness (mm)	Force (N)	Failure stress (MPa)	Yield strength (MPa)
CuFe-1	3.40	0.057	28.5	148	82
CuFe-2	2.84	0.05	31.5	172	67
CuFe-3	3.64	0.05	30.6	169	66
			Average	163	72
Cu-1	3.16	0.052	3.4	92	40
Cu-2	4.00	0.052	3.8	81	31
Cu-3	3.67	0.052	3.39	82	38
			Average	85	36

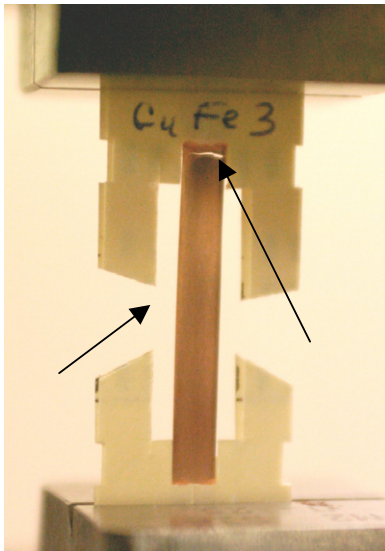


Figure 19. Photograph of the biaxially textured Cu–Fe specimen (Cu–Fe3) at the end of the test. Notice that the separation distance of the cut cardboard has increased as compared to figure 3.

generated. The crosshead displacement throughout the test and at failure was used to determine the specimen elongation since the grip and cardboard compliance was much lower than

the actual foil specimens. Figure 19 shows the specimen at the end of the test, showing the typical fracture observed in all of the tensile tests. Notice the separation distance of the cut cardboard as compared to figure 3 showing the total elongation. Once the specimen had failed, elastic recovery caused the crack opening displacement space to be small. Since there was an insufficient quantity of material to test as a function of gauge section length to account for the test system compliance, as prescribed in ASTM D3379, the actual elastic modulus was not determined. In all tests, the specimens failed within the cardboard gauge section cutout and as such are considered valid tests, as prescribed in ASTM D3379. The yield strength was determined using the 0.2% offset strain method, as prescribed in ASTM E-8 (standard test method for tensile testing metallic materials). Thus, the yield strength values can be used comparatively within this study.

Figure 20 shows the pseudo stress–strain curve of one set of Cu–Fe and Cu samples. The average yield strength of the Cu–Fe alloy samples was found to be 72 MPa, whereas for pure copper samples it was found to be 36 MPa, indicating that these Cu–Fe alloy substrates are at least twice as strong as the copper substrates. Table 4 shows the tensile test data taken from all the three sets of Cu–Fe and Cu samples along with the sample dimensions, showing that the samples showed consistent results in terms of yield stress and fracture stresses in this test. The increased yield strength of the Cu–Fe alloys

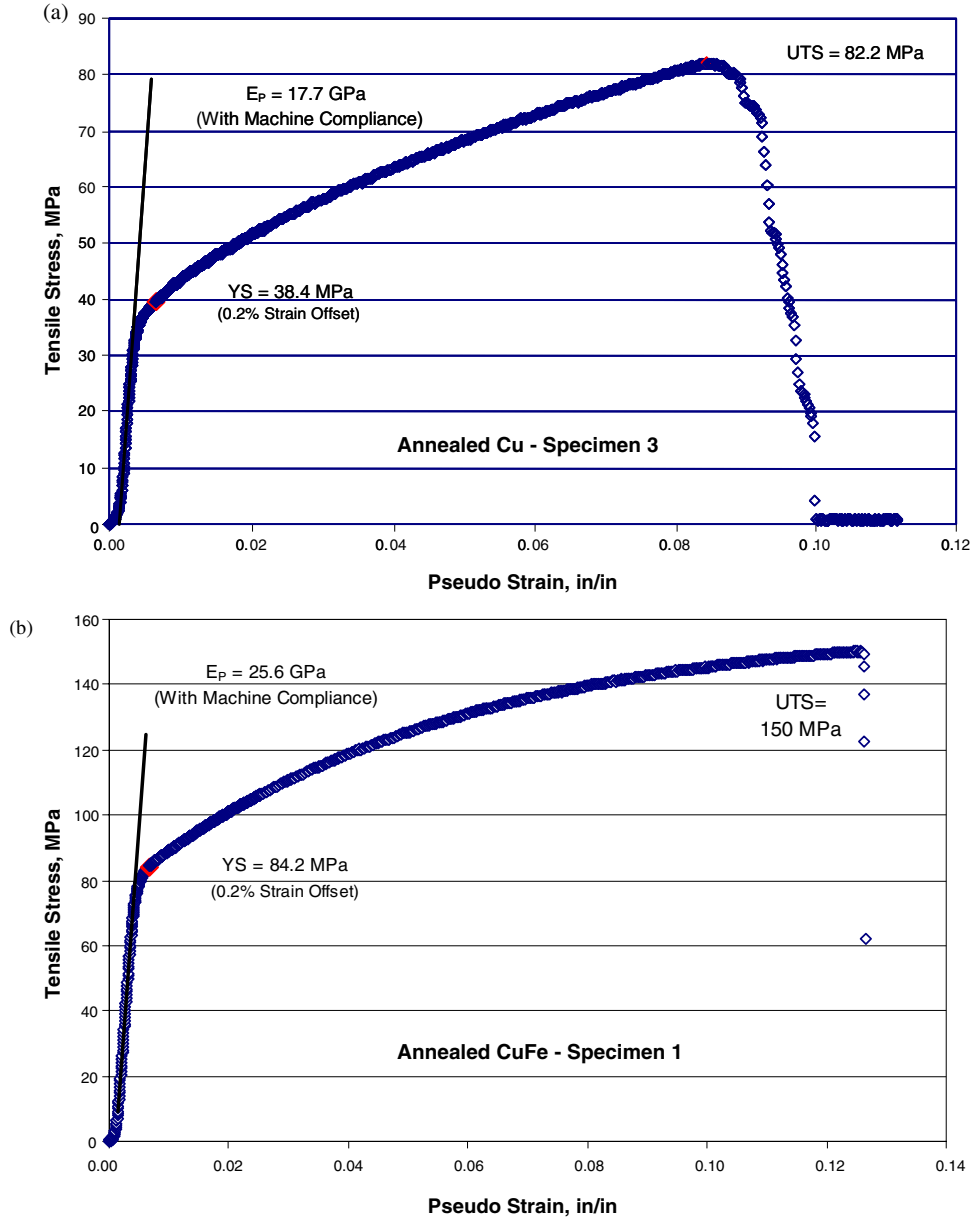


Figure 20. The stress–strain curves of (a) Cu and (b) Cu–Fe samples.

is expected to be beneficial during the fabrication of coated conductors. Use of these substrate materials will allow a greater ease in handling of the conductor during processing.

Figure 21 shows a scanning electron micrograph of a Cu–Fe sample. It can be seen that $\sim 0.5 \mu\text{m}$ -sized precipitates are present on the surface of the Cu–Fe samples. These were found to be Fe-rich Cu-alloy precipitates and are thought to be potentially responsible for the increase in the strength of these alloys through a dispersion hardening mechanism. The presence of other additives such as P and Zn was also observed besides Fe in these precipitates. Even though the alloying elements are less than 2.5 wt%, since the precipitates are Cu-alloy-based precipitates, the number density of the precipitates is observed to be high.

The effects of these precipitates on the quality of the texture of buffer layers and YBCO are not known at

present. Experiments are presently underway to understand the formation mechanism and effects of these precipitates. It may be possible by proper selection of heat treatment to control the size and distribution of these precipitates and create a textured template with two dimensional defects. Effective construction of that template can potentially propagate the defects beneficially into the subsequent epitaxially grown films and serve as flux pinning regions for the superconductor. Initial results [17] of Ni–20% Cr layer deposition on the Cu–Fe substrates showed that biaxially textured buffer layers can be grown on these substrates, indicating that the surface of the substrate between the precipitates is a clean and lattice-matched surface.

Although the pure copper demonstrated slightly better texture, the texture of the Cu–Fe is adequate for producing quality YBCO-coated conductors. The primary concern is

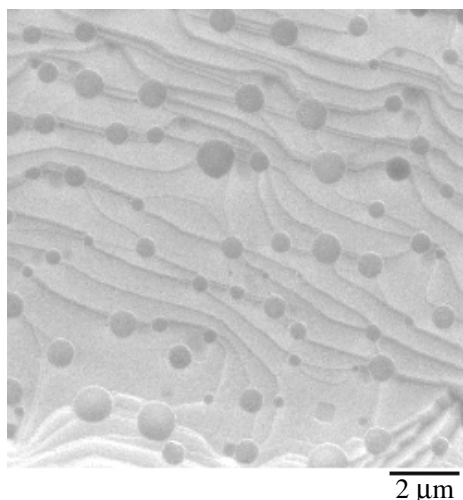


Figure 21. A scanning electron micrograph of a textured Cu-Fe sample showing the presence of fine precipitates.

the presence of the precipitates on the surface of the Cu-Fe substrates. Oxidation of the potentially Fe-rich precipitates should be of no greater concern than the Cu itself. Indeed a buffer stack has been demonstrated for pure Cu and oxidation of the substrate can be mitigated by metallic plating [16], sputter coating of the substrates [18, 19] or by using TiN buffer layers [7]. The critical requirements for replacement of Ni-W are strength, magnetic properties, and electrical resistivity. Cu-Fe alloys meet two of three metrics, i.e., the magnetic properties and electrical resistivities are comparable to or better than Ni-W. Even though the yield strength is higher than that of annealed Ni, it is still lower than that of Ni-W for Cu-2.35% Fe alloy at room temperature. Further improvements may be possible with higher amounts or additional alloying elements.

4. Conclusions

Pure copper and copper-iron alloy metallic substrates with very good biaxial texture have been prepared by optimizing the thermo-mechanical treatments. An average FWHM of ϕ scans of 5.5° was obtained on copper samples and 5.9° on a Cu-Fe sample. The magnetization data on Cu-Fe samples showed values that were low compared to pure nickel samples and comparable to Ni-W alloys currently used. The resistivity of the Cu-Fe sample was found to be higher than that of pure Cu at 77 K. The yield strength

of the Cu-Fe sample is also found to be better than that of pure Cu samples by at least a factor of two. A Cu-alloy will likely be necessary for implementation of copper-based substrates in lieu of the Ni-based substrates presently used.

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