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FINE GRAINED Nb for INTERNAL TIN Nb_3Sn CONDUCTORS

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ABSTRACT

The push to drive superconductor strand technology to reach higher critical current density (J_c) values and reduce production costs has led to innovative approaches in manufacturing technology. The Restacked Rod Process (RRP®) by Oxford Instruments is one such process which involves Nb bar extrusions in a Cu sheath. Commercially available Nb used in the initial RRP extrusion leads to nonuniform deformations of the Nb bar which in turn leads to a jagged Cu-Nb interface. This report presents a feasible methodology to remedy the problem of nonuniform deformation of Nb through severe plastic deformation (SPD) of precursor Nb to obtain smaller grains in starting Nb. Cu-Nb monocore extrusion and drawing experiments were accomplished at Oxford Instruments using Nb bars with grain sizes in the range of µm to mm. Results of Cu-Nb interface roughness measurements show that a finer starting grain size gives a significantly lower roughness and better Nb core conformance to initial shape. Our experiments indicate that refinement of the initial Nb grain size to below ~50µm could enable fabrication of RRP conductor with improved wire yield and higher J_c.

KEYWORDS: Cu-Nb, fine grain Nb, high J_c, RRP, ECAE, SPD, interface roughness.

INTRODUCTION

At present Nb_3Sn stands are used in many of the best performing high field magnets [1]. With the successful development of the internal tin (IT) RRP® at Oxford Instruments in 2001[2], J_c has been pushed to higher levels than were possible with other methods. RRP involves Nb rods hot extruded in a Cu sheath. This approach offers significant advantages:

a) better control over Cu to Nb ratios and hence Cu interfilament distance and b) easy scalability. This paper presents a possible approach for the manufacture precursor Nb rods with better microstructure.

The performance of Nb_3Sn strand is directly dependent on the Cu:Nb:Sn ratios. From the works of Peter Lee and others [1, 3-10], it is seen that high Sn:Nb ratios are beneficial for the formation of a homogeneous distribution of the Nb-Sn A15 compound. Obtaining the right ratios of Cu:Nb:Sn involves a complex interplay between the Nb strand size, amount of Cu , architecture of the wire, and the manufacturing process. One of the challenges presently faced by RRP conductors is instability at low fields which has been attributed to the large Nb sub-element size [11, 12]. This problem is worsened by non uniform subelement deformation and sub element merging during wire drawing[13].

Understanding deformations between adjacent Cu-Nb regions during the initial fabrication steps is beneficial in realizing methods to lessen Cu-Nb interface deformations and obtain a better roundness for the Nb strands in a Cu matrix. Better deforming Nb will improve predictability of the fabrication process so that subsequent sub element deformation non-uniformities can be decreased. The authors suggest microstructure control through thermo mechanical processing of precursor Nb as a strategy to obtain better Cu-Nb codeformation. Proof-of-concept experiments with Cu-Nb monocore wires demonstrate the feasibility of the idea.

MATERIALS AND METHODS

I. Starting Material and Processing of Precursor Nb.

The starting Nb used for three of the four monocore experiments was as-cast grade I Nb, also called reactor grade (RG), supplied by ATI Wah Chang. The composition being 99.9% Nb, 1500 ppm Tantalum (Ta) and trace amounts of carbon, hydrogen, nitrogen and oxygen. The as-cast blocks were cut to nominal dimensions of 50mm x 50mm x 250mm using a wire EDM. The Nb used for the fourth monocore was provided courtesy H.C. Stark. This grade I bar had a grain size of 1-5mm. The Cu sheath used for all monocore was commercially available OHFC Copper, in conformance with ASTM F68-05.

To effectively break down the as-cast structures in two Nb bars thermomechanical processing was employed. This involved refining the microstructure by severe plastic deformation (SPD) through multi pass equal channel angular extrusion (ECAE)[14] and subsequent recrystallization heat treatments. This process has been previously demonstrated to effectively refine as-cast Nb[15, 16]. Table 1 provides details of the starting Nb bars used in the mono core experiments. The change in chemistry of the H.C. Stark (#11050) material is reflected by the relatively higher hardness compared to the other samples. To obtain different grain sizes, the as-processed Nb was heat treated at different temperatures: $950^{o}C$ for the finer grain size (#11051) and $1150^{o}C$ for the larger grain size sample (#11052). Figures 1(a-d) show the representative macro/micro structure for the starting Nb samples.

Table 1. Characteristics of the starting Nb bar for the monocore experiment.

Monocore ID Number	**Starting Nb bar characteristics**		
	Processing	**Grain Size**	**HV300**
11049	As cast	10-40 mm	54.1 ± 2.1
11050	As received from HC Stark	1-4 mm	63.5 ±3.3
11052	Multi pass ECAE and recrystallized @1150C	70-170 µm	55.9 ±1.2
11051	Multi pass ECAE and recrystallized @ 950C	20-60 µm	57.2 ±1.5

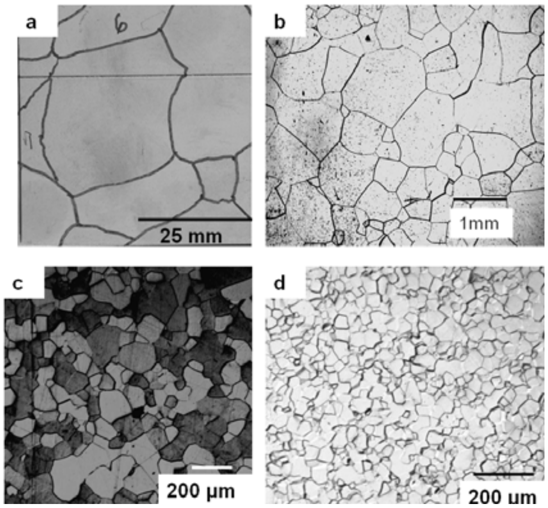

Figure 1(a-d). Representative microstructures of the starting Nb used in the fabrication of Cu-Nb monocore filaments. a) Cast sample with a grain size of 10-40mm;b) H.C. Stark Nb, average grain size of 1-4mm; c) ECAE processed and heat treated at 1150^{o} C, average grain size 70-170µm. and d) ECAE processed and heat treated at 950^{o}C, average grain size of 20-60 µm.

II. Monocore fabrication

The starting Nb rods were nominally 45mm in diameter and 78mm long. These rods were placed into an OHFC Cu conduit of outer diameter 51mm, evacuated, and end plug welded. The Cu-Nb composite was then warm extruded to an outer diameter of 16mm. The extruded samples were drawn down by 15-20% reduction per pass. The final wire diameters to which the Cu-Nb monocore wires were drawn to was 1mm. Samples were taken from intermediate drawing stages for characterization and analysis.

III. Post Processing Analysis

The wire samples were hot mounted in Bakelite, mechanically polished, and optical micrographs taken using a Leica DM500 optical microscope. The copper on the wires was removed, using a 10%HNO_3 solution, exposing the Nb core, and the longitudinal surfaces were examined using an FEI Quanta 600 FE-SEM. To get a quantitative measure of the roundness or conformance of shape of the Nb core, the standard deviation (SD) of the copper layer thickness was determined for each wire. To normalize the roughness value the SD was divided by the wire outer diameter.

RESULTS

The Cu-Nb interface roughness first appears in the warm extruded bars as shown in Figure 2 (a-d). The jaggedness of the interface or deviation from roundness appears to be greatest for the Nb with the largest starting grain size.

The Cu-Nb interface roughness increases slowly initially and at an increased rate as the wire is drawn down. Figure 3 plots of normalized roughness. If we assume the initial roughness of 0.01 as a reference, then the increase in roughness between the warm extruded and final drawn wire size is 600% for the sample with the starting initial grain size of 10-40mm and drops to 80% for the sample with smallest grain size. Figure 4(a-d) give an indication of how Nb plastically deforms as it has been extruded and drawn. The longitudinal ridges are typical of extruded and drawn wires sheathed in a softer material.

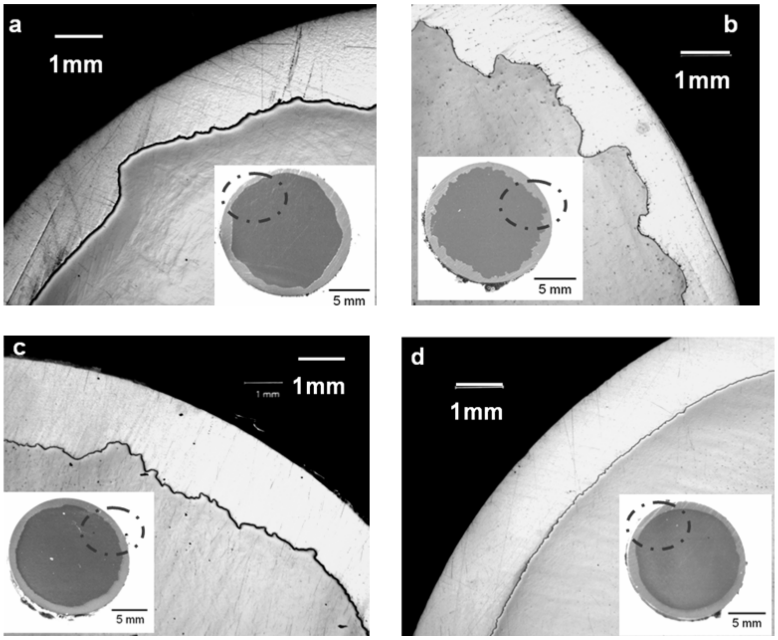

Figure 2(a-d). Representative sections of the Cu-Nb interface of warm extruded monocore rods with grain sizes - a) 10-40 mm (#11049) ;b) 1-4 mm(#11050) ;c) of 70-170µm (#11052); d) 20-60 µm (#11051).

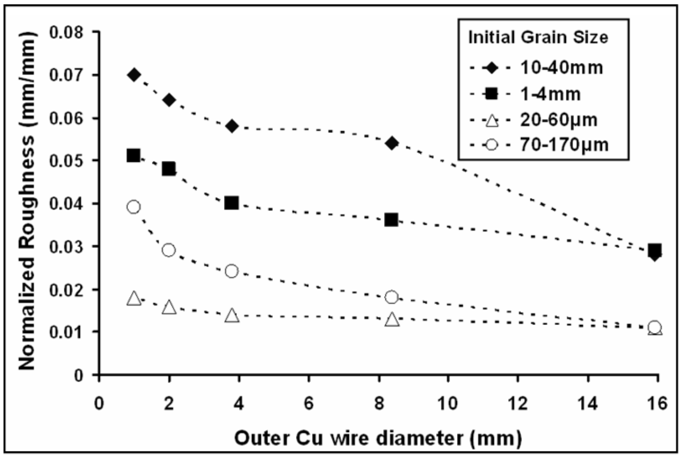

Figure 3. Roughness ratio as a function of wire diameter for wires with different initial Nb core starting grain sizes.

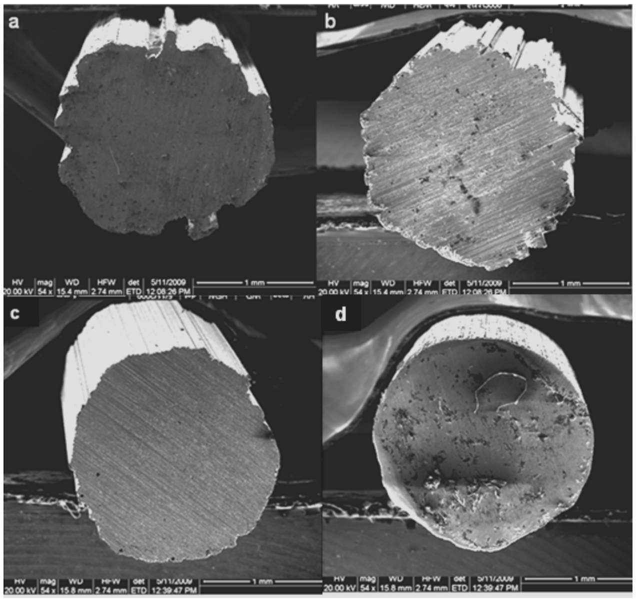

Figure 4(a-d). As drawn Nb wire section, with the Cu sheath etched off: a) sample #11049, initial grain size 10-40mm, b) sample#11050, initial grain size 1-3mm , c) sample #11052, initial grain size70-170µm , d) sample #11051, initial grain size 20-60µm.

DISCUSSION

To analyze the roughness phenomenon, the process of roughening can be broken down into several parts: a) creation of instability, b) instability reaching a critical value, and c) propagation of the instability and worsening of interfacial roughness. Thilly et. al suggested that during the drawing of Cu-Ta interfaces, the creation of roughness depends on the instability due to differences in the shear modulus between interfacial surfaces in contact [17, 18]. The shear moduli of Cu and Nb are 46 GPa and 37.5 GPa, respectively. However, there may be other causes such as load eccentricities and mechanics of the fabrication operation which may play a major role in the initial creation of these instabilities. These instabilities cause stable features at the interface [17]. In this regard the state of stress of the body could play an important role too. Once a stable feature is created further deformation propagates and worsens the feature which appears as the macroscopic interface roughness.

Our interest also lies in the worsening of roughening features. The authors suggest that the propagation of the roughness is based on the inelastic components of the deformation and microstructure. The main component of the roughness stems from the attributes of the microstructure especially grain size and texture. The initial hot extrusion process seems to be the critical step, after which the roughness once created only worsens. The yield behavior, after the hot extrusion process for two different wires is very similar as the material is drawn down. This is shown in Figure 5 where it can be seen that the hardening behavior of the wires follows a similar trend. This indicates that the plastic deformation character, especially the yielding and hardening behaviors are similar for the best and the worst wire after the initial hot extrusion. Differences in this behavior however may arise in the individual wires during the initial stages of deformation. Further experiments need to be done to confirm if a variation in mechanical property exists.

In general any mechanical processing operation reorients the grains to favorable orientations which show up as the texture of the material after processing [19]. In the presence of applied stresses, the major deformation of the grains is in the form of stretch and rotation. The differences in the deformation of the different grain shows up as the roughness on a free surface, which is the interface in our case. This would mean that the bigger the grain and the more unfavorable the initial orientation, the larger will be the deformation and bigger the displacement left behind on the surface. Clearly, the Cu-Nb monocore wire results indicate that the initial grain size has a major influence on the deformation and interface roughness character. Figures 6(a-c) show a rendering of the microstructure of a polycrystalline material with a free surface undergoing large strain deformations.

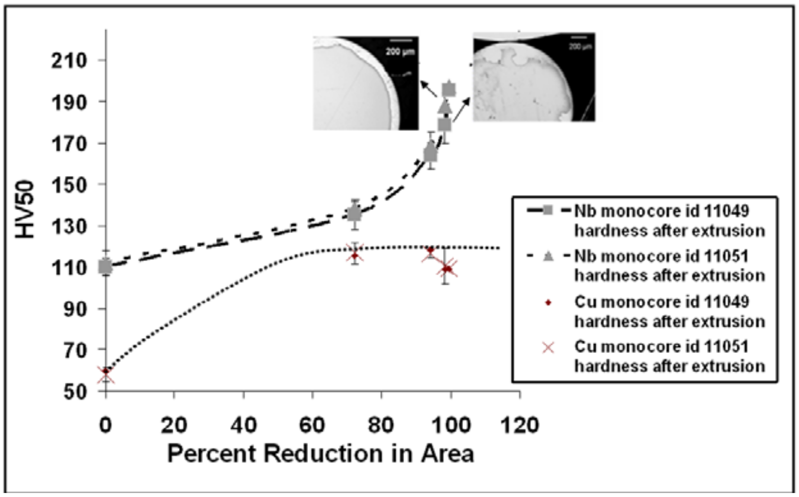

Figure 5. Hardness versus percent reduction in area for the best and worst wires indicating the change in yield strength and hardening behavior as these wires were drawn down.

The authors hypothesize that initial textures play an important role in non-uniform deformation. Nb being a bcc material, has the major slip components in the temperature range under consideration of {110}<111> [20]. The extrusion and drawing textures in Nb are predominantly the {110} textures, which are widely reported in the literature [21-23]. Our best Cu-Nb results were obtained with the finer grain size (20-60 µm) ECAE processed sample. The authors believe that the ECAE processed sample has the most favorable microstructure because of a uniform and fine grain size and favorable texture components, predominantly the {110} planes aligned in the extrusion direction. Further experiments involving the intermediate stages of hot extrusion along with microstructural characterization could confirm this hypothesis and lead to a better understanding of the deformation mechanics in Cu-Nb composites.

SUMMARY AND CONCLUSIONS

1. A finer initial Nb grain size leads to better conformity of roundness of Nb rods in a Cu matrix.
2. The initial hot extrusion process is important for achieving better Nb uniformity and deformation characteristics.
3. Favorable microstructures for Cu-Nb codeformation by warm extrusion and wire drawing can be developed by ECAE.
4. Future work needs to focus on the effects of texture along with the grain size to determine the most optimum microstructure.

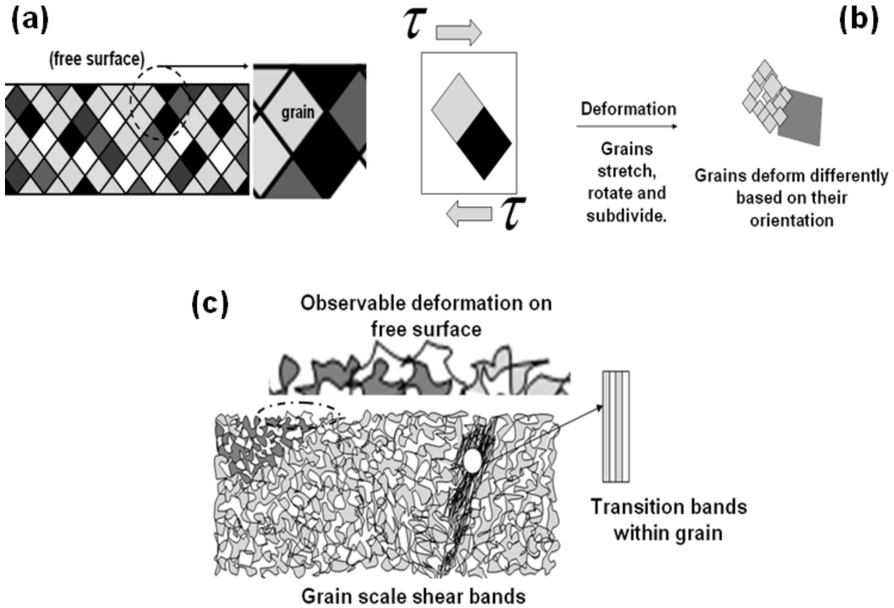

Figure 6. a) A polycrystalline material with square grains. The different shades of gray represent different textures. b) As the sample is deformed, internally the grains deform by rotating and stretching and eventually refine into smaller grains. The sub dividing and rotating process depends on the initial orientation of the grain. c) Macroscopic view after deformation with the appearance of the surface roughness including the presence of shear bands on the surface.

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