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**Production of Seamless Superconducting Radio Frequency Cavities
from Ultra-fine Grained Niobium**

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Executive Summary

The positron and electron linacs of the International Linear Collider (ILC) will require over 14,000, nine-cell, one meter length, superconducting radio frequency (SRF) cavities [1]. Manufacturing on this scale will benefit from more efficient fabrication methods. The current methods of fabricating SRF cavities involve deep drawing of the halves of each of the elliptical cells and joining them by high-vacuum, electron beam welding, with at least 19 circumferential welds per cavity. The welding is costly and has undesirable effects on the cavity surfaces, including grain-scale surface roughening at the weld seams. Surface roughness limits accelerating fields, because asperities prematurely exceed the critical magnetic field and become normal conducting. Hydroforming of seamless tubes avoids welding. This method has been pursued at DESY in Hamburg, Germany, using 150 mm diameter tubes produced by extrusion or spinning from plate [2-4]. Extruded tubes exhibited poor formability, due to strong textures and texture banding. Tube from plate showed a range of structure and properties from nose to tail. Both types of tube developed surface roughness during hydroforming due to large grain sizes.

Schematic diagrams of single cell and 9-cell SRF cavities of the ILC geometry

This project explored the technical and economic feasibility of an improved processing method for seamless tubes for hydroforming. Severe deformation of bulk material was first used to produce a fine structure, followed by extrusion and flow-forming methods of tube making. Extrusion of the randomly oriented, fine-grained bulk material proceeded under largely steady-state conditions, and resulted in a uniform structure, which was found to be finer and more crystallographically random than standard (high purity) RRR niobium sheet metal. A 165 mm inch diameter billet of RRR grade niobium was processed into five, 150 mm I.D. tubes, each over 1.8 m in length, to meet the dimensions used by the DESY ILC hydroforming machine. Mechanical properties met specifications. Hydroforming and superconducting testing will be pursued in subsequent collaborations with DESY and Fermilab.

The economic feasibility was demonstrated. The production cost of the prototype tubes was slightly more than twice that of RRR niobium sheet. This cost should decrease with volume production. The five prototype tubes were processed at three geographically distinct locations, and some scrap loss was needed for tuning of the flow-forming parameters. A realistic estimate places the cost of large volumes of tube on par with the cost of RRR sheet, which is currently about \$240/lb. The cost differences between the welding and hydroforming fabrication methods then amounts to the relative cost of final assembly. Hydroforming of seamless tubes is more amenable to mass production than high-vacuum electron beam welding and significantly cheaper. The scientific community

and the general public will benefit by adoption of this process since costs of production will be lower and the availability of accelerator cavities will improve. SRF Cavities are used for other areas of high-energy physics research, as well as for medium and low energy particle accelerators, and in Free Electron Lasers. These have applications in the fields of basic science, industrial processing, medical diagnostics, pharmaceutical research and weapons systems.

Comparison of accomplishments with the goals and objectives of the project

Objective:

The objective of this project was to develop the enabling technology for efficient hydroforming production of ILC, SRF cavities. The intermediate goals were: 1) to develop a severe deformation processing method to produce a fine structured, bulk, 165 mm diameter RRR niobium billet, 2) to extrude the billet to make 150 mm diameter tubes with a fine microstructure and uniform, axisymmetric properties and 3) to hydroform the tube into ILC specification SRF cavities at the DESY facility in Hamburg, Germany, and determine their SRF properties.

This was a significant undertaking for two reasons. First, seamless tubes of RRR niobium of this size had not been made before in the U.S and second, it is difficult to maintain a fine grain size in pure metal when it is subject to several sequences of strain and heating. Tubes of this size have been manufactured in Germany, but the properties have left room for improvement. Tube extruded from large grained billets had large grains, elongated structures with a strong crystallographic alignment of grains and texture banding. The interior surfaces developed severe surface roughening and formability was poor. Surface roughening during plastic straining may occur due to intrinsic factors and manifest as dislocation glide steps, shear bands, orange peel, and larger scale deformation patterns arising from the co-deformation of clusters of similarly oriented grains (often of a single texture component). The connection between roughness with strain and grain size is well established [5, 6]. Recent studies have examined the relationships between texture and roughening [7-11], and texture “clustering” and roughening [10]. Researchers at DESY evaluated tube made from plate to take advantage of the benefit of strain processing of the plate to break up the coarse grain billet [2]. This material, however, showed a large range of grain structures and properties along the tube length, which is undesirable, since uniform and consistent properties are important for hydroforming.

The novel approach of this project was to use severe deformation and carefully chosen thermal treatments to produce a fine structure in bulk material which *remained stable* through subsequent processing. Severe deformation (greater than a true strain of about 4), is not possible during standard metalworking processes, except for processes yielding thin gage material [12]. Cyclic strain methods, e.g. equal channel angular extrusion (ECAE), torsion processing and multi-axis forging, allow high strains to be developed in the bulk material, since the volume of material is returned to the same shape during subsequent cycles.

Preliminary work was done on small-scale specimens, to select the billet processing conditions. A 165 mm diameter billet was required for the 150 mm diameter extrusions, and this was produced from RRR niobium and processed by the selected method. The subsequent work was to compare the tube microstructure, properties and strain-roughening behavior to other tube and sheet; and the final goal was to hydroform the tube into ILC specification SRF cavities at the DESY facility in Hamburg, Germany, and determine their SRF properties.

Results:

Billet Processing. The Phase I project demonstrated the feasibility of using equal channel angular extrusion (ECAE) to produce a very fine grain structure in RRR niobium billet. Ultrafine-grained material (i.e. $< 1 \mu\text{m}$ diameter grains) was not produced. This is likely due to: a limit of true strain achieved to less than 7 or 8; the lack of any second phase, grain growth limiting particles in the high purity; and the presence of a large area fraction of low angle boundaries. Finite element method estimates of the forming pressures required for processing a full-size, 165 mm diameter billet in the ECAE process, with an added 50% overhead safety margin, actually exceeded the maximum limit of the available 3500 ton press.

We also evaluated several, alternative, proprietary, severe strain processes, in collaboration with ATI Wah Chang (AWC). This option was specified in the Phase II work plan. The alternative processes examined produced fine, more random and more stable billet microstructures than those that we obtained by ECAE. After comparison of variants of several methods, we selected one of the alternate processing routes which provided superior results in intermediate scale (50 mm diameter) tests, with lower forming pressure. This alternate method was adopted for scale-up. Scale-up to a 165 mm diameter billet was successful and proceeded to tube production.

Tube Production. Tube production involved back-extrusion, forward-extrusion, flow-forming and recrystallization. Of the five, 150 mm I.D. x 1.5 m length tubes for which processing was attempted, two were needed to adjust the flow-forming conditions, and were not produced with uniform wall thicknesses per specifications. The three tubes meeting specifications were recrystallized and prepared for hydroforming.

Comparisons were made between samples of the finished tube, RRR niobium sheet, and samples provided by DESY from earlier tube making attempts. Specimens were ranked for formability by DESY, and that information was combined with the microstructural observations to predict the relative formability of the BL/AWC tube. Study of the DESY tube showed that the best correlation between microstructures formability were the extent of recrystallization and the randomness of crystallographic texture. The BL/AWC tube showed better recrystallization, a weaker texture and also, a much smaller grain size than the other materials.

Mechanical property tests were conducted on samples cut from tube, and included longitudinal and circumferential tensile tests. Samples of the BL/AWC tube were compared to some “spun from plate” [2] DESY material and to RRR niobium sheet. The tests for all specimens exceeded the minimum, 40% ductility, requirements stated by DESY. The BL/AWC tube showed higher strength levels, consistent with their smaller grain sizes (Hall-Petch behavior), and both microstructures and properties were consistent from tube to tube. Surface roughening profilometer measurements were performed on the DESY material (spun from plate) and the BL/AWC tube. These comparisons showed a five-fold improvement in the tube interior surface roughness for the finer grained BL/AWC tube.

Hydroforming and Testing. Our collaborators at DESY agreed to perform both the hydroforming and SRF testing on the hydroformed cavities. However, changes were made in the processing schedules due to research results on the intermediate-scale tests, and these delayed tube production. The DESY hydroforming equipment was not available by the time the final tubes were produced, since the workforce at DESY had been re-allocated to higher priority tasks.

The intermediate goals of the project have been met. Very-fine grained RRR billet material was produced, and the fine grain size was retained through tube production and was found to be consistent from one tube to another. The crystallographic texture of the tube was weaker than either standard sheet or any of the previously produced DESY tube. This suggests better formability.

The final objective, of SRF testing on a hydroformed SRF cavity, awaits forming and testing at DESY. Tube has been delivered to DESY for these tests. The first hydroforming tests of this tube are scheduled to be performed at DESY under the auspices of a Fermilab program, and will constitute a Phase III effort. SRF testing will be performed on successfully hydroformed cavities.

Summary of Project Activities

The Phase I project demonstrated the feasibility of using equal channel angular extrusion, ECAE [13, 14], to produce a remarkably fine grain structure in RRR niobium billet. The Phase II Work Plan sought to: develop a grain refining method by ECAE or an alternate process so that an extremely fine, random and stable microstructure could be developed in a 165 mm (6.5”) diameter billet; produce the refined billet and use it to produce 150 mm (4”) tube; form the tube into ILC SRF cavities; and test the SRF properties of the hydroformed cavities. Two assumptions were made in the planning stages of this work: 1) that ECAE could be scaled to a 165 mm diameter extrusion billet, within the time and financial constraints of the program and 2) that a submicron (ultrafine) grain structure would be needed in the billet stage to produce a structure comparable to RRR sheet (i.e. a ~50 μm grain size) in the final tube. Both assumptions were wrong, but without any detrimental consequences. The first was deemed impractical for several reasons. The second, fortunately, was not necessary for the microstructure developed in this program.

An alternate, high-strain, process was developed which resulted in production of a billet with a uniformly fine structure which remained stable through the production of the tube. Several entities collaborated in this project, and their names are listed below, along with the abbreviations which will be used in the rest of this report.

ATI Wah Chang, Albany Oregon	AWC
Black Laboratories, L.L.C., Newport News, VA	BL
Deutsches Elektronen-Synchrotron, Hamburg, Germany	DESY
Florida State University, Tallahassee, FL	FSU
Thomas Jefferson National Accelerator Facility, Newport News, VA	JLab

The specific tasks of the Work Plan are shown below.

1. Selection of ECAE die geometry.
2. ECAE processing of 6.5" billet.
3. Preparation of a second billet by an alternate process.
4. Back Extrusion, Forward Extrusion – Flow forming of 150 mm tubes.
5. Hydroforming of Cavities at DESY.
6. Determination of Cavity performance by SRF Testing at JLab/DESY.

The procedure for these tasks followed a flow chart sequence, as shown in Figure 1. Experiments were performed during each of the completed tasks to determine the conditions for the subsequent process. Extensive use is made of orientation imaging mapping (OIM) [15, 16] for analysis of the metal microstructure and various stages of processing (OIMTM is a trademark of Edax, which is a unit of Ametek Materials Analysis Division, of the Ametek corporation). OIM maps are produced by collecting a grid of electron backscattered diffraction (EBSD) pattern solutions and comparing the orientations, the statistics of the orientations (in orientation distribution functions, or ODF's) and spatial variations of the orientations (e.g. by inverse pole figures), and the point-to-point orientation changes (expressed in grain boundary misorientation plots or grain orientation spread maps). The term "texture" refers to a high frequency of grain orientations in the orientation distribution, and is measured here by the maximum intensity of the ODF calculation. Other experimental techniques used included finite element method simulations with LS-Dyna, and tensile tests and profilometer measurements of surface roughness.

Task 1. Selection of ECAE die geometry

High levels of work, near a true strain of 4, can only be produced in conventional processing by significant reduction in thickness of the workpiece, e.g. in rolling of sheet metal to thin foil [12]. High strains can be imparted to large workpieces by cyclic methods, including equal channel angular extrusion (ECAE), torsion, or cyclic-multi-axial forging. An early decision for the production of seamless tube was to take advantage of steady state conditions during extrusion to produce tube with axisymmetric properties. This required a 165 mm (6.5") billet for the production of 150mm (6") diameter tube. Processing of such a large billet by the severe deformation method of

ECAE requires a well designed, robust die, and a large extrusion press. Preliminary experiments in Task 1 were performed to assess the viability of this concept with the 90° elbow, circular cross-section die used in Phase 1. The selected ECAE process, known as Bc, uses a 90° rotation between extrusion passes, in one rotation direction. The die provides approximately a true strain of 1 per pass. The 90° die, with a circular cross-section and a curved corner was built and tested for 0.5 inch (12.7 mm) diameter billets (Figure 2). Dies were constructed of hardened, 4340 steel, and assembled with 0.75-16 bolts. A 3.25 inch (83 mm) length ram was used to push the billet in a servo-hydraulic test frame. Die channels were polished to a mirror finish, and engine assembly lubricants were used between contact surfaces. An expanded channel was used at the exit, to minimize drag of the elastically expanding metal. Galling and welding developed at the niobium and steel interface. The dies were repolished and coated with a titanium nitride layer. Galling remained, however, as shown in the striations in the die-locked billet in Figure 3a, which stopped at a forming pressure of 896 MPa (130 ksi). A finite element analysis confirmed the much higher pressures at the die elbow (Figure 3b), which led to galling. Experience with copper billets led to sheathing of the niobium with copper tube, and this allowed complete extrusion at 717 MPa (104 ksi).

Seamless SRF Cavity Development

Figure 1. Flowchart depicting sequence of tasks and decision process for selection of final processing parameters.

Figure 2. ECAE die schematic for 1.27 cm (0.5 inch) diameter RRR niobium billets. Billets were placed in the top of the die and extruded through the side.

Figure 3. ECAE extrusion billet and die pressure: a) bare niobium locked in die, note striations due to galling; b) LS-Dyna simulation of pressure distribution in die, note high pressure point at elbow, where the most galling occurs.

Subsequent experiments demonstrated the effectiveness of using aluminum-bronze inserts in the die, rather than copper sheathing.

Figure 4. Successful ECAE extrusion of copper-sheathed niobium billet.

The microstructure of ECAE billets were fine-grained, but showed a stronger than expected tendency for preferred orientation. The color proximity of grains in an inverse pole figure map, Figure 5, suggests a strong texture, which was confirmed by the maximum ODF intensity of 19.

Figure 5. OIM Inverse Pole Figures, showing crystal directions normal to the plane of polish and revealing the effect of ECAE on grain size, for: a) forged starting stock, b) 1 pass ECAE, c) 4 passes ECAE (true strain ~ 4) + 700°C recrystallization anneal.

Figure 6. Grain Orientation Spread, GOS, for the ECAE processed material shown in Figure 5c. Recrystallized grains are selected as those with less than 1° GOS, which cover less than 7% of the area examined.

The OIM results are summarized in Table 1.

Table 1. OIM Results of RRR Nb

Condition	Grain Size, μm, 10° boundary	ODF Maximum Intensity	% Recrystallized, based on GOS ≤ 1°
Forging	110	39	82
1 Pass ECAE	51	20	< 1
4 Pass ECAE + Anneal	15	19	7

Task 2. ECAE processing of 6.5" billet

Processing studies of scaled-up billets were limited to simulations. Although extrusion was possible at pressures of more than 700 MPa (>100 ksi), this is approximately an order of magnitude higher than the yield strength of niobium 70 MPa (~10 ksi). There was significant flashing between the die halves with the 1.27 cm (0.5 inch) diameter billet

(see above the elbow in Figure 4). Improvements such as hydraulic joining clamps were considered as possible die design improvements.

Simulations were run in LS-Dyna for 1.27 cm, 5.8 cm and 16.5 cm (0.5, 2 and 6.5 inch) diameter billets at 25, 400 and 700°C working temperatures. A standard industrial die friction coefficient of 0.1 was assumed for a commercial process. The higher temperatures, either 400 or 700°C, would result in a reduction in ram force of about 20%, if extrusion at high temperatures were feasible.

Results for the 165 mm (6.5 inch) diameter billet are shown in Figure 7. The pressure needed for extrusion at 25°C is large. The maximum ordinary ram force for the 165 mm (6.5 inch) billet at 25°C is expected to be about 22 kN (~2500) tons. That is the value without any unexpected problems in the die or extrusion billet which may raise the force requirement. AWC personnel indicated a need for a 50% pressure overhead, as a safety margin. This would necessitate an extrusion press capacity of 3750 tons, which is greater than the 3500 ton press available.

The results of these simulations led to the decision to limit further work to the right-hand side of the decision tree in Figure 1, and pursue development of the ATI Wah Chang alternate process. The ECAE process had been evaluated as a means of producing an ultra-fine, submicron, grain structure, which was expected to give a better chance of retaining a fine structure throughout the tube forming process. As it turned out (and as shown in the following task descriptions), the alternate process produced a finer and more stable structure, which remained stable throughout the process. The decision to avoid the ECAE approach had no negative impact on the results. The positive impact was shown in the feasibility of scale-up.

Figure 7. Ram force (kN) (a) and strain distribution (b) for ECAE of a 165 mm (6.5 inch) diameter billet. Force requirements stabilize near 23 kN, or about 2500 tons.

Task 3. Preparation of a second billet by an alternate process

The alternate process was developed in collaboration with AWC. A systematic study was conducted by examination of the microstructures of RRR niobium processed by several different routes. The goal of the billet processing was to produce a microstructure which would facilitate the production of axisymmetric tube with a fine grain structure. This was considered to require the following characteristics: a fine grain size, a weak texture and a fully recrystallized condition with a low volume fraction of substructure. The final requirement has been found to be important in severe strain processing, and metals and alloys with more than a 30% area fraction of low-angle grain boundaries (with angles $< 10^\circ$) are not stable and likely to undergo anomalous grain growth during annealing. The low angle boundaries are a potent force for coarsening [12]. The microstructures which remain stable after the recrystallization anneal are more likely to retain a fine structure during the subsequent extrusion processes, which are performed at an elevated, but lower temperature than the recrystallization temperature (although with applied strain).

Intermediate scale, high strain processing was performed on samples of 50 mm (2 inch) diameter RRR niobium at AWC, with a BL employee in attendance. Multiple processing variants were evaluated for scale-up, and each variant was examined after several different, final, recrystallization heat treatments. The samples were examined by optical microscopy and evaluated by hardness testing at AWC, to estimate the grain size and amount of recrystallization. Three selected conditions were then studied by orientation imaging microscopy (OIM) at FSU, to determine texture, true grain size and area fraction of low angle grain boundaries.

OIM inverse pole figure maps for variants A, B and C are shown in Figures 8, 9 and 10, for samples from the outside surface (surface), quarter (t/4) and mid (t/2) sections of the processed billets. Grain sizes were calculated, and the percent of recrystallization was determined by the area fraction with $GOS \leq 1^\circ$.

Figure 8. OIM inverse pole figure maps of Process A. Note the bands of similar grain

colors. The corresponding average grain sizes in μm were 12, 9.1, and 6.3 and the recrystallization fractions were 91%, 86% and 86%.

Figure 9. OIM inverse pole figure maps of Process B. Selected process. The corresponding average grain sizes in μm were 12, 11, and 11 and the recrystallization fractions were 97%, 96% and 92%.

Figure 10. OIM inverse pole figure maps of Process C. Outside grain coarsening. The corresponding average grain sizes in μm were 8.9, 6.0, and 5.1 and the

recrystallization fractions were 88%, 90% and 94%.

Figures 8, 9 and 10 alone show that process B has the most uniform grain size, and a fairly random grain orientation distribution. Process A has the finest interior grain size, but the most coarsening towards the outside of the billet, as well as an apparent occurrence of texture banding (shown as the mostly red color in the upper left side of Figure 8a). Process C has both randomness and a fine structure, but shows excessive coarsening on the outside. Stability is related to the presence of low angle grain boundaries (or substructure). The amount of substructure can be measured by a plot of grain boundary length as a function of angle. Figure 11 shows these plots for the t/4 section for processes E (ECAE), A, B and C.

Figure 11. Number fraction vs. Misorientation Angle of grain boundaries from data sets for a) Process A (t/4), b) Process B (t/4), c) Process C (t/4) and e) ECAE.

The results for each data set are summarized in Table 2, and compared to those for the ECAE. Results include grain size, ODF maximum intensity, the percent of recrystallized material, and the percent of low-angle grain boundaries.

Table 2. OIM Results of Billet Processes

	A, surf.	A, t/4	A, t/2	B, surf.	B, t/4	B, t/2	C, surf.	C, t/4	C, t/2	E, t/2
Grain size, μm	12	9.1	6.3	12	11	11	8.9	6.0	5.1	15
ODF max.	6.3	17	20	4.4	7.3	8.2	23	9.1	7.1	19
% Rx	91	86	86	97	96	92	88	90	94	7.0
% lagb	6.8	10	10	2.9	7.8	14	10	7.5	5.3	64

Figure 12. Texture intensity (ODF max) from the data sets for Processes A, B and C.

Figure 12 shows that the texture intensity, expressed as the maximum ODF intensity, is the most uniform for Process B. Process B was selected for scale-up to 165 mm diameter.

Task 4. Back Extrusion, Forward Extrusion – Flow forming of 150 mm tubes

A 165 mm (6.5 inch) diameter billet of RRR grade, high-purity niobium was processed according the conditions designated Process B. The billet was recrystallized, back-extruded and forward extruded at AWC to a 12.7 mm (0.500 inch) wall thickness pipe. It was then shipped to Dynamic Flow Form in Billerica, Massachusetts for flow forming to final dimensions with a wall thickness of 3.5 mm (0.138 inches) and lengths of 1.5 m (~60 inches) or greater.

The tube was then returned to AWC for pickling (acid cleaning) and recrystallization anneal. Images of the tube sections are shown in Figure 13.

Figure 13. 150 mm (6 inch I.D.) tubes after flow-forming. The white bands on tubes B1A and B2A indicate wall thickness changes during flow-forming set-up.

An important part of this task was the determination of parameters to judge the success of the processing variations. This was approached by evaluating the microstructures of previously prepared material, including standard RRR niobium sheet, three samples of tube prepared for the DESY machine, and standard, commercial RRR niobium sheet. The DESY tube material was from a lengthy study of various processing methods and was provided as part of the collaboration. There were some questions as to the microstructures and relative properties of the tube: the extruded tube had very poor formability; the spun-from-plate tube was better in the nose than in the tail, and all showed greater surface roughening during plastic deformation than did sheet. The results of the comparisons of these attributes and the acceptable formability of RRR sheet suggested that recrystallization and a weak texture were most important. For the current project goal, we have added the requirement of a fine grain size to give lower surface roughening.

Figure 14. AWC RRR niobium sheet. a) Inverse Pole Figure (IPF); b) GOS Map.

RRR sheet was obtained from AWC for determination of standard property microstructures, since the sheet is used for deep drawing of half-cells for standard e-beam welding assembly of SRF cavities. The corresponding inverse pole figure is shown in Figure 14. The inverse pole figure (IPF) shows the randomness of the structure (by the distribution of colors). A different OIM map, the grain orientation spread (GOS), was used to show the extent of recrystallization. A spread of 1° or less was chosen as the definition of a recrystallized grain. From the plot in Figure 14b it is seen that the sheet metal was 92% recrystallized in the region examined. The average grain size was measured at $25\ \mu\text{m}$, and the ODF maximum at 14.

Figure 15. DESY Extruded and flow-formed tube. a) IPF, b) GOS map.

Figure 15 shows the IPF and GOS maps for the DESY extruded material. The IPF map shows significant “texture banding”, which is evident by the similar colors in three regions of the map. The grains are also large, compared to the sheet metal (Figure 14). The material was only 75% recrystallized, as seen in the GOS map in Figure 15b, and more than 6% of the grains showed an internal misorientation range of more than 3°. Through wall thickness maps were used to determine the average grain size of 50 μm , and the ODF maximum at 33.

Figure 16. DESY Tube spun from plate and flow-formed. OIM maps: a) IPF, nose; b) IPF, tail; c) GOS, nose, 90% recrystallized; d) GOS, tail, 82% recrystallized.

The two samples of one tube spun-from-plate show a large grain size variation from the nose (Figure 16a) to the tail (Figure 16b). The spinning process appears to impart greater strain to the tail region, which receives more cold work, with a resulting smaller recrystallized grain size. The nose reportedly showed better formability, even though it contains some deformed grains (e.g. the red grain in the lower left of the GOS Map in Figure 16c), it had better overall recrystallization. Through wall thickness maps were collected for both of these tube pieces. The average grain size for the nose was measured at 500 μm , and the ODF maximum at 15. The average grain size for the tail was measured at 100 μm , and the ODF maximum at 17.

Figure 17. OIM images of BL/AWC tube B1B. Second of five through-thickness OIM scans: a) an image quality map, showing the grain shape; b) an IPF map, showing the randomness of grain orientations; and c) a GOS map, showing the distribution of recrystallized and deformed grains. The recrystallized fraction from the GOS map was 90%.

Through wall thickness maps of the BL/AWC tube, B1B, were used to determine the average grain size of 14 μm , and the ODF maximum at 7 (Figure 17). There was some change in texture through-thickness, but it remained weak throughout. The microstructures of tubes B1B and B2B were very similar.

The BL/AWC tube had the smallest grain size, the weakest texture. The recrystallization was comparable to the RRR sheet and DESY tube spun-from-plate.

These results are summarized in Table 3.

Table 3. OIM data for Sheet and Tube, from Through-Thickness Measurements

Condition	Grain Size, μm , based on 10° boundary	ODF Maximum Intensity	% Recrystallized, based on $\leq 1^\circ$ GOS
Sheet	25	14	92
DESY Extruded + flow- formed	50	33	75
Spun-from-Plate + flow- formed, nose	500	15	90
Spun-from-Plate + flow- formed, tail	100	17	82
BL/AWC Extruded + flow-formed	14	7	90

Task 5. Hydroforming of Cavities at DESY

Results from the intermediate-scale tests at ATI Wah Chang suggested changes in the processing schedule and related delays. These full-scale prototype components were manufactured in an industrial facility and scheduling changes required considerable delays. The DESY hydroforming equipment was not available when the final tubes were produced, since work at DESY had been re-allocated to higher priority tasks.

Mechanical property tests are required prior to hydroforming, and were conducted as part of this task. Longitudinal and circumferential tensile tests were performed in the U.S. for the BL/AWC tube and compared to sheet and samples of DESY tube, and to RRR sheet metal obtained from Jefferson Lab. Tests were conducted according to ASTM E-8, for subscale specimens. Both Longitudinal and circumferential tests were performed on samples of material flattened from tube sections.

Table 4. Summary of Mechanical Property Data

Sample Name	Orientation	Yield Strength, MPa (ksi)	Elongation, %
J-A	Unknown	79.3 (11.5)	60
D-T	Circumferential	65.5 (9.5)	59
B1B-T	Circumferential	84.1 (12.2)	51
B1B-L	Longitudinal	84.8 (12.3)	55
B2B-T	Circumferential	84.8 (12. 3)	52
B2B-L	Longitudinal	82.7 (12.0)	61

Some of the tensile samples were examined on the tube interior side to compare surface roughening resulting from plastic deformation. Photographs of three of the samples are shown in Figure 18.

Figure 18. Macrophotographs of surface roughness of tensile samples, from Table 4, pulled to fracture. Note rough surface on the D1TA sample, which is the large grained material shown in Figure 16a.

The BL/AWC tube showed higher yield (12.3 ksi) strength than the RRR sheet (11.5 ksi), which was also higher strength than the DESY tube (9.5 ksi). This may be explained by grain size differences, due to the phenomenon known as Hall-Petch behavior, where the yield strength σ_y increases at a rate proportional to the inverse square root of the grain size:

$$\sigma_y = \sigma_0 + K_y d^{-1/2}$$

and where d is the grain size, σ_0 is a minimum stress, and K_y is a constant termed the Hall-Petch slope. The average grain sizes of the three materials are 14, ~50 and 500 μm , respectively. Plotting the yield strength as a function of the inverse square root of the grain size (Figure 19) shows a good fit to a linear, Hall-Petch relationship with a $\sigma_0 = 62.9$ MPa, a slope, K_y , of 87.0, with a linear regression coefficient of determination, $R^2 = 0.95$. A perfectly linear relationship would result in an R^2 value of 1.

Figure 19. Trendline and yield strength results showing Hall-Petch relationship showing increase in yield strength as a function of the inverse square root of the grain size. Linear regression, coefficient of determination, $R^2 = 0.95$.

The higher yield strength for the fine-grained tube is explained by Hall-Petch behavior, i.e. it is due to the finer grain size. If this behavior holds to much smaller grains, it is worth noting that a grain size of $1\mu\text{m}$ would show a yield strength of 150 MPa, which may have been an issue for a hydroforming system designed for 70 MPa. The surfaces appear quite different. The larger grain material shows significant roughness, while the small grained sheet and BL/AWC tube (B1B) surfaces are much smoother. The difference in roughness between the B1B tube and the large-grain DESY tube were measured in a Dektak profilometer, the results of that study are shown in Figure 20. These comparisons showed a five-fold improvement for the BL/AWC tube.

Figure 20. Surface roughness measurement data from tensile samples using a Dektak Profilometer. Vertical axis is $\text{k}\text{\AA}$ ($= 0.1\mu\text{m}$). Horizontal axis is μm , with a full scale of 1 cm. Comparison of DESY tube (spun from plate) and

BL/AWC tube, showing a five-fold improvement for the fine grained BL/AWC tube.

Tube has been delivered to DESY, Figure 21. Measurements at DESY show the tube, B2B, meets wall thickness variations, except for a few points at 0° in the first 50 cm, which were attributed to surface scratches.

Figure 21. BL/AWC RRR Nb tube, B2B a) in DESY Lab in Hamburg; b) DESY measurement of thickness profile of tube at 0° , 90° , 180° and 270° . Thickness variation better than 0.100 mm for most of length; deviation at 0° due to surface scratches. Courtesy Andy Hocker, Fermilab.

Hydroforming is scheduled within a few months, and will use the equipment shown in Figure 22.

Figure 22. The DESY hydroforming facility in Hamburg: a) the hydroforming press, with pre-spun irises on copper test cavities; b) close-up showing ILC hydroforming dies mounted on the press.

Task 6. Determination of Cavity performance by SRF Testing at JLab/DESY

This effort was transferred to DESY early in the program. DESY personnel agreed to perform the testing at DESY after completion of the cavity hydroforming. SRF testing will be performed on successfully hydroformed cavities, and will be a part of the program continuation under Fermilab sponsorship.

Products developed and technology transfer activities

The purpose of this project was to develop a new product, namely a fine-grained, high-purity, RRR niobium tube capable of being hydroformed to the geometry of the ILC SRF cavities. Profilometer measurements on tensile tests showed that the fine grain structure achieved in this material resulted in significant, five-fold reductions in surface roughening over previously manufactured types of seamless tube. This is predicted to result in better SRF performance than earlier tube and at a significant cost savings over standard electron beam welding fabrication methods.

Production of this product required the development of a new processing technology for RRR niobium, which was achieved under this award. Collaborations were developed under this award and included partnerships with ATI Wah Chang, DESY, Fermilab and Jefferson Lab. A new and improved seamless, RRR niobium tube technology has been developed during this program and will enable the efficient, large-scale production of SRF cavities.

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