Effect of thermo-mechanical processing on the material properties at low temperature of a large size Al-Ni stabilized Nb-Ti/Cu superconducting cable

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Abstract. For future high-resolution particle experiments, a prototype for a 60 kA at 5 T, 4.2 K class conductor is realized by co-extrusion of a large, 40-strand Nb-Ti/Cu superconducting cable with a precipitation type Al-0.1wt.%Ni stabilizer. Microalloying with nickel contributes to the strength of the stabilizer, and avoids significant degradation in residual resistivity ratio, owing to its low solid solubility in aluminum.

Sections of the conductor are work hardened to increase the mechanical properties of the as-extruded temper. Mechanical and resistivity characteristics are assessed as function of the amount of work hardening, at room temperature as well as at 4.2 K.

Thermal treatments, like resin curing after coil winding, can cause partial annealing of the cold-worked material and reverse the strengthening effect. However, targeted thermal treatments, applied at relatively low temperature can result in precipitation hardening. The depletion of nickel in the aluminum-rich matrix around the precipitates results in an increased strength and a decreased effect of nickel on the thermal and electrical resistivity of the material.

The present work aims at identifying an optimal work hardening sequence, and an optimal thermal treatment, possibly coinciding with a suitable coil resin curing cycle, for the Al-Ni stabilized superconductor.

Keywords: aluminum stabilized superconductor, Al-Ni alloy, mechanical properties, work hardening, thermal treatment, ultrasonic measurement
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INTRODUCTION

Recent proposals for multiple high-energy physics experiments, like CLIC, ILC, Mu2e, and IAXO, include high-current, large-bore size superconducting magnets for the analysis of the angular momentum of charged particles [1]. The increase in energy range in these experiments often call for the increase in both magnetic volume as well as magnetic field for acquiring useful particle trajectories. In the large, often solenoidal magnets, carrying a large quantity of electric current, a high hoop stress will be developed in the windings. Due to the self-supporting nature of the magnet structure, the conductor needs to sustain the high mechanical stress and corresponding strain, which requires for it to exhibit challenging mechanical properties.

In the high-resolution particle detectors which utilize a superconducting cable to realize a high magnetic field, a stabilizer is required around the superconducting cable to protect the superconductor by the diversion of the electrical current and withdrawal of heat when the superconductive state locally returns to a normal conductive state due to a perturbation [2]. High-purity aluminum, of a purity of 99,999% or more, is being used as a stabilizer material in many composite conductors for high-energy physics magnets, rather than copper [3]. Aluminum compares favorably with copper because of its high transparency to particles, its excellent thermal and electrical conductivity at 4.2 K, and its lower cost. High-purity aluminum is characterized by its high residual resistivity ratio (RRR= $\rho_{293K}/\rho_{4.2K}$, where ρ =resistivity), however also by its low mechanical strength.

The necessity for the development of a large size, reinforced aluminum-stabilized superconductor is apparent. A method for strengthening high-purity aluminum, allowing a reasonable preservation of conductivity characteristics, is dilute precipitation alloying. A high-purity aluminum matrix is retained by preventing impurity solute atoms in solution, allowing a high RRR. In the Al-Ni system, nickel and aluminum form non-coherent intermetallic compounds already at room temperature, due to the low solid-solubility of nickel in aluminum [4]. The process of compound formation by depletion of nickel in the aluminum-rich matrix can be enhanced by a thermal treatment known as age hardening. The precipitation hardening of high-purity aluminum with nickel leads to both dislocation pinning by the Al_3Ni compounds as well as by extended dislocation retainment by grain boundaries due to grain size decrease with nickel alloying [5]. A dilute Al-0.1wt%Ni alloy was developed for the ATLAS central solenoid conductor [6, 7, 8]. This alloy achieved a yield strength of ∼70 MPa at room temperature and a RRR of ∼600 after a cross-section reduction of ∼20% due to cold drawing, for a 30 mm x 4.3 mm plated conductor.

In a previous study by the authors a process scale-up has been realized by the co-extrusion of a large, 40-strand Nb-Ti/Cu superconducting cable with the precipitation type Al-0.1wt%Ni alloy, to a record cross-section size of 57 mm x 12 mm [5]. The experimental results of the work-hardened conductor suggested further study. A yield strength 25% lower than that of Al-0.1wt%Ni stabilizer in the ATLAS central solenoid conductor was measured, leading to the cautious conclusion that increased cross-section extrusions result in decreased work-hardening effects.

In the follow-up of this study presented here, the conductor is subjected to work hardening by single-pass and limited-pass industrial bi-directional rolling. Subsequently two work-hardened states are subjected to 15 h heat treatments at various temperatures, from which an optimal coil resin curing treatment is determined.

MATERIALS AND EXPERIMENTAL METHODS

For the present study, a unit length of 20 m co-extruded Al-0.1wt%Ni stabilized superconductor was available for further assessment of mechanical and resistivity properties as function of its mechanical and thermal treatment, sufficient for the measurements performed. The parameters of the co-extrusion process are described in [5].

Work Hardening by plastic deformation is applied to increase the mechanical properties of the as-extruded temper. A production-scale experiment was performed, using 1.5 m long sections of the co-extruded Al-0.1wt%Ni stabilized superconductor. The aim was to confirm the properties of cold rolled co-extruded Al-0.1wt%Ni on a production-scale, to determine the optimal area reduction, and to assess the workability by bi-directional rolling as function of the amount of passes. Work hardening was applied on a 50 ton DEM, actively driven, four-roll Turks-head mill at Criotec, Chivasso (I); an installation also used for the ITER cable-in-conduit conductor production [9, 10]. Force was applied mainly in the short transverse direction (ST), while the wide transverse direction (TD) was constrained to preserve cable integrity and maintain a realistic aspect ratio (width/thickness). The samples are rolled in contra-extrusion direction to account for spooling and despooling in the case that the work hardening takes place at a different location from the extrusion. Cold rolling was applied both in single step as well as in multiple steps, to determine material characteristics with work hardening sequence. Moreover, one sample was subjected to a homogeneous reduction, by equal reduction in ST direction as in TD direction, to evaluate bonding characteristics with work directionality.

Mechanical Measurements: Specimens for tensile testing were removed from the center of the aluminum-based stabilizer part of the conductor with their long axis parallel to the extrusion direction (LD), similar to previous studies [5]. The specimens were carefully machined by spark erosion to a 25 mm gauge length, and a 3 x 3 mm^2 cross-section to minimize additional work hardening. Room temperature measurements were conducted on an electromechanical universal tensile machine at a constant stress speed of 5 MPa/s up to one half the specified yield strength, and subsequently slowed down to a deformation speed of 1.5 mm/min according to ASTM standard E8/E8M.

A setup developed by CERN for high-precision tensile measurements at 4.2 K was utilized for cryogenic tensile testing [11]. Specimen, analogous to the RT specimen, immersed in a liquid helium cryostat, were tested using the same universal tensile machine and custom made extensometers. Stress-strain curves were initially determined by measuring at a stroke rate of 0.5 mm/min (congruent with a strain rate of $\sim 3.3 \cdot 10^{-4}\ s^{-1}$), for subsequent specimens stroke rate was decreased to 0.15 mm/min (strain rate $1 \cdot 10^{-4}\ s^{-1}$), both consistent with ASTM standard E1450-09. At least three measurements are conducted on each tensile measurement serie.

RRR Measurements, here defined as the ratio of resistance measured at 293 K and 4.2 K, are performed with the experimental setup described in [12]. Specimens are machined with use of spark erosion in the longitudinal direction (LD) to a length of 110 mm, voltage tap length of 80 mm, and a 2 mm x 2 mm cross-section. During mounting of the RRR specimens, care is taken not to deform the thin specimens to avoid additional work hardening. At least two specimens are measured for each material variant.

Quality of Bonding: For good performance of the conductor a proper void-free bonding between the copper matrix based strands of the Rutherford cable and the aluminum-based cladding must be ensured. This will provide the

TABLE 1. Properties of co-extruded Al-0.1wt%Ni as a result of various cross-section reductions due to bi-directional rolling

	Temp. [**K**]	**RRR**	$\mathbf{R}_{p0.2}$ [**MPa**]	$\mathbf{R}_{m}$ [**MPa**]
As-extruded	293	1191	26	53
	4.2	-	57	303
20% single pass cold-rolled	293	656	62	67
	4.2	-	127	376
30% single pass cold-rolled	293	404	75	81
	4.2	-	157*	496

* deduced from two measurements

superconducting cable with an effective heat and current transfer in case of the development of a normal zone, and will ensure mechanical stability. In the co-extrusion process cable cleaning by brushing and cable preheating were adopted to assure the best quality of bonding [13].

Ultrasonic Measurements: Subsequent to co-extrusion and work hardening, extensive studies on the quality of bonding are performed at the Swiss Federal Laboratories for Materials Science and Technology (Empa), on both broad sides of the conductor over short lengths (200 mm pieces). Mechanical C-scanning, applying a pulse-echo immersion technique, is used to reveal detailed information on the bond-quality [14]. A 20 MHz ultrasonic probe scans the surface of the short sample while it is immersed in demineralized water. The resulting C-scan is a two-dimensional plot of the echo amplitudes, between 0 mV and 1000 mV, of the stabilizer-superconductor interface, where the various amplitudes are indicated by a different color. The quality of bonding is deduced from the amplitude, a small amplitude corresponds to a good bonding and a large amplitude to a poor bonding.

X-ray Radiography: In addition to ultrasonic testing, the samples were measured with an X-ray micro computed tomography (CT) setup installed at Empa. For the initial radiographic images a Viscom XT9160-TDX source was operated at 140 kV and 0.07 mA and the emitted spectrum was hardened with a 0.1 mm thick lead filter and detected by a Perkin Elmer XRD 1621 CN3 ES detector. Subsequent measurements were done by using a Viscom XT9225-TEP X-ray source with an acceleration voltage of 200 kV and a 1 mm thick copper hardening filter to improve the contrast of the Rutherford cable. A more detailed description of the measurement setup and image reconstruction can be found in [15].

Thermal Treatment: Short lengths of cold-rolled Al-0.1wt%Ni stabilized conductor have been submitted to various thermal treatments in a SNOL 9/1100 furnace, and mechanically tested in order to assess the stability of the mechanical properties against resin curing cycles subsequent to coil winding. 20% and 30% cold-rolled short samples have been subjected to 15 h lasting thermal cycles, congruent with typical resin curing cycles. Temperatures were adopted between 373 K and 630 K, to determine lattice defect recovery temperature while staying within reasonable temperature range for resin curing. The temperature near the samples was monitored with a SEFRAM 7337 multimeter, the temperature reached a variation of ± 4.5 K at the lowest temperature, and only ± 1 K at the highest temperature.

Indentation hardness measurements are performed on the thermally treated samples with a Wolpert Wilson universal hardness tester. The hardness measurements (HV) employed a Vicker's pyramid diamond indenter at 10 kgf load (HV10), applied for 10 - 15 s, to obtain an approximate 0.8 mm diameter indentation, in accordance with ISO standard 6507-1. Hardness values were obtained from the center of the aluminum-based stabilizer, to eliminate possible skin effects due to cold rolling. The hardness values reported here are determined from at least six individual measurements for each material condition.

RESULTS

Significant increase in Mechanical Properties. Table 1 summarizes the average results of the main properties for the co-extruded Al-0.1wt%Ni stablizer for different cross-section reductions by cold rolling. Results are reported for the as-extruded state, 0% cold worked, and for the 20% and 30% in cross-section reduced states, at both room temperature as well as cryogenic temperature. At room temperature, the Al-0.1wt%Ni stabilizer in the reference as-extruded state exhibits the lowest value of 0.2% yield strength, $R_{p0.2}$, of 26 MPa, as to be expected. For the 20% cold-reduced case a significant average increase is observed to an $R_{p0.2}$ of 62 MPa. The subsequent increase in $R_{p0.2}$ from the 20% cold-reduced case to the 30% reduced case (75 MPa) is less distinct. The evolution of the ultimate tensile strength, R_m of the co-extruded Al-0.1wt%Ni material with cross-section reduction follows a more continuous behavior from 53 MPa in the as-extruded state, trough 67 MPa for the 20% cold-reduced case, to 81 MPa for the 30% reduced case. For the cold-reduced cases $R_{p0.2}$ approaches R_m, however, for this particular application, $R_{p0.2}$ is the leading property for success or failure of the conductor. The strong increase in $R_{p0.2}$ of the as-rolled states compared with the as-extruded state confirms a high workability of the Al-0.1wt%Ni material in a production-scale work-hardening sequence.

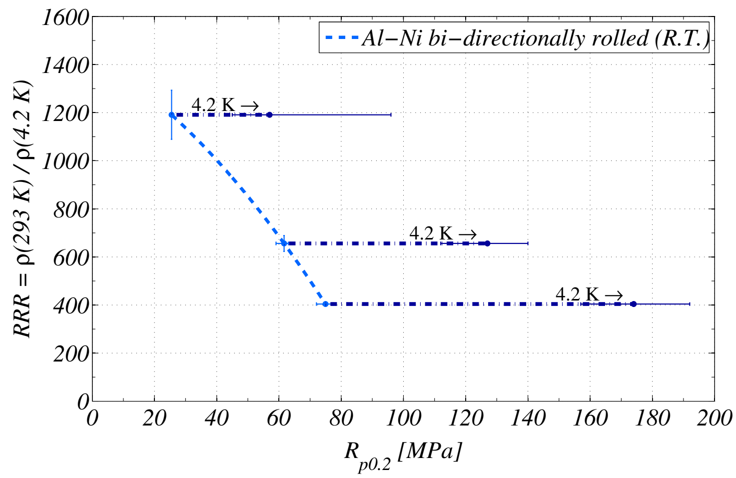

FIGURE 1. RRR plotted as function of $R_{p0.2}$ for the various cold-worked states, at both cryogenic as well as room temperature tensile measurement temperature. Notice the large increase in $R_{p0.2}$ at cryogenic temperature with respect to room temperature.

The increase in tensile properties is quite significant from a measurement temperature of 293 K to 4.2 K for the various cold-rolled states (Table 1 and Fig. 1). All as-rolled states present an $R_{p0.2}$ above 100 MPa at 4.2 K, accompanied by an R_m above 350 MPa. The improvement of mechanical properties at cryogenic temperature result in an average $R_{p0.2}$ of 57 MPa for the as-extruded state, and an average of 127 MPa and 174 MPa $R_{p0.2}$ for the respectively 20% and 30% cold-reduced states. A good correlation is found between $R_{p0.2}$ at cryogenic temperature and at room temperature for all material states, the ratio ($R_{p0.2}$ at 4.2 K / $R_{p0.2}$ at 293 K) was found to be between 2.1 and 2.3.

Fig. 1 shows the deterioration of RRR with increasing cross-section reductions as result of cold-rolling. The RRR falls off from an average 1191 for an as-extruded state to an average of 404 for a 30% cold-reduced state. A roughly linear interaction between RRR and $R_{p0.2}$ at room temperature is reported for the co-extruded Al-0.1wt%Ni stabilizer material. This linear relationship also holds for the $R_{p0.2}$ at cryogenic temperature, although for this case a less steep descent of RRR with increase in $R_{p0.2}$ is visible. From the average results in Fig. 1 it can be noticed that the mechanical and resistivity properties of Al-0.1wt%Ni can be modified by mechanical processing to fit the requirements for a 60 kA critical current at 5 T class conductor, exhibiting a >120 MPa $R_{p0.2}$ at 4.2 K, while preserving a RRR of more than 500. When extrapolating the results at 4.2 K, an Al-0.1wt%Ni stabilizer exhibits an $R_{p0.2}$ of 120 MPa at 4.2 K, when cold-reduced by ∼19%, while still maintaining an RRR of ∼700.

Degraded quality of Bonding. Ultrasonic measurements on the short samples show to give important information on the quality of bonding of the Al-Ni based stabilizer to the copper Rutherford matrix subjected to various work-hardening cycles. Fractions of the short samples measured by ultrasound are shown in Fig. 2, accompanied by X-ray radiography images of the same fractions. In the ultrasonic images, subfigures a1, b1, c1, d and e, delamination of the Al-Ni based stabilizer is marked by a high amplitude echo, bright colored area. In the X-ray radiography images, subfigures a2, b2 and c2, a light colored area indicates strand breakage. Fig. 2a1 and a2 show a fraction of a short sample of the 15% reduced conductor. In the center of this short sample (displayed fraction) no delamination is revealed by ultrasonic measurement, moreover no strand breaking is indicated by radiography. On the contrary, for the 35% by cold-rolling reduced case, major delamination can be observed (Fig. 2b1). This delamination is accompanied by strand breakage, which can be witnessed in Fig. 2b2. It appears that due to a large strain on the delaminated strands,

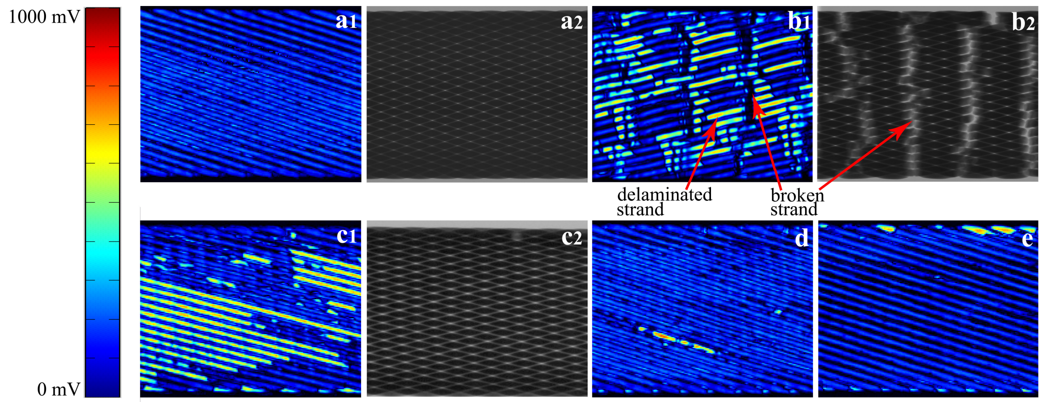

FIGURE 2. Ultrasonic C-scans and X-ray radiography images of co-extruded, cold-rolled Al-Ni stabilized conductor. Subfigures *a1*, *b1*, and *c1* show C-scans of 15, 35 and 20% one pass cold-rolled conductor, respectively. Subfigures *a2*, *b2* and *c2* show the corresponding X-ray radiography images. Subfigures *d* and *e* show C-scans of 20% cold-rolled conductor in two passes, and homogeneously reduced, respectively.

by not having the support of the attached stabilizer material, the strands break. The locations of strand breakage were consistent with concave ripples present on the Al-Ni based stabilizer surface, indicating flow of stabilizer material into the void where no strand is present.

In Fig. 2c1 a C-scan of the 20% cold-worked sample is presented, accompanied by its radiographic image in Fig. 2c2. Major delaminations are present, and some minor indications (top right) of strand cracking (light colored) are found in the radiography images. There are, however, only a few marginal marks present. Further study on a longer lengths should indicate whether a reduction of 20% by single-pass cold-rolling results in only delamination or if the strand breakage, observed here, occurs as well.

Response to Partial Annealing. In Fig. 3 the effect of a thermal curing cycle on 20% and 30% cold-rolled conductor specimen can be observed. The diagram is based on hardness measurements on short specimens subjected to 8 different thermal treatments for 15 h in the range between room temperature (293 K) and 630 K. The diagram clearly indicates a temperature range where recovery of lattice defects takes place, reversing the work-hardening effect, and resulting in lower mechanical properties. One can observe a distinct difference in recovery temperature for the different cold-rolled states. Recovery is mainly situated between 470 K and 530 K for the 20% cold-worked state, whereas for the 30% cold-worked state the recovery already takes place between 450 K and 510 K. For both states no indication of precipitation hardening effects can be distinguished. This precipitation hardening due to artificial aging, however, is unlikely to occur due to the low diffusivity of Ni in Al at these relatively low temperatures [16].

DISCUSSION

At room temperature the deviation of individually measured values from the mean values is minimal. However, from Fig. 1, at 4.2 K, the deviation from the average values is quite significant. A factor that could account for the relatively large spread in obtained values is a strain rate sensitivity of the Al-0.1wt%Ni material. Tensile specimens were tested at two strain rates; $3.3 \cdot 10^{-4}\ \mathrm{s}^{-1}$ and $1 \cdot 10^{-4}\ \mathrm{s}^{-1}$. The higher strain rate resulted in slightly higher mechanical properties, indicating that the Al-0.1wt%Ni material is strain rate sensitive at cryogenic temperature. The results presented in the current study indicate a very promising average 2.1-2.3 ratio of $R_{p0.2}$ at 4.2 K over $R_{p0.2}$ at room temperature. A ratio in line with other aluminum alloys, e.g. aluminum alloy 6061, which exhibits a $R_{p0.2}$ at 4.2 K / $R_{p0.2}$ at 293 K ratio of 1.8 - 1.9 in a T4 temper state [17]. A slight inconsistency of the results obtained here with the results obtained on the Al-Ni material in the development of the CMS and ATLAS solenoid conductors, reporting a $\sim$1.5 ratio [18**?**], could therefore be the result of the material's strain rate sensitivity due to a different measurement speed. Further cryogenic tensile study at a strain rate similar to what is experienced by the material in operational situation, can give a decisive answer on this topic.

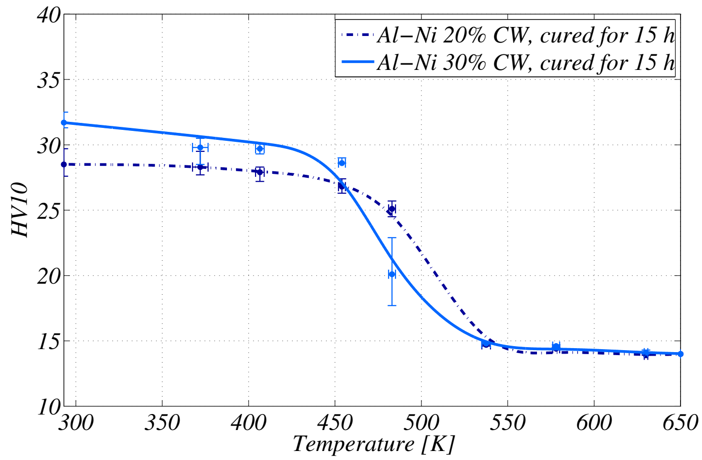

FIGURE 3. Hardness, HV10, plotted as function of thermal treatment temperature. The data presents HV10 values of 20% and 30% single pass cold-rolled short samples subjected to various thermal treatments with a duration of 15 h.

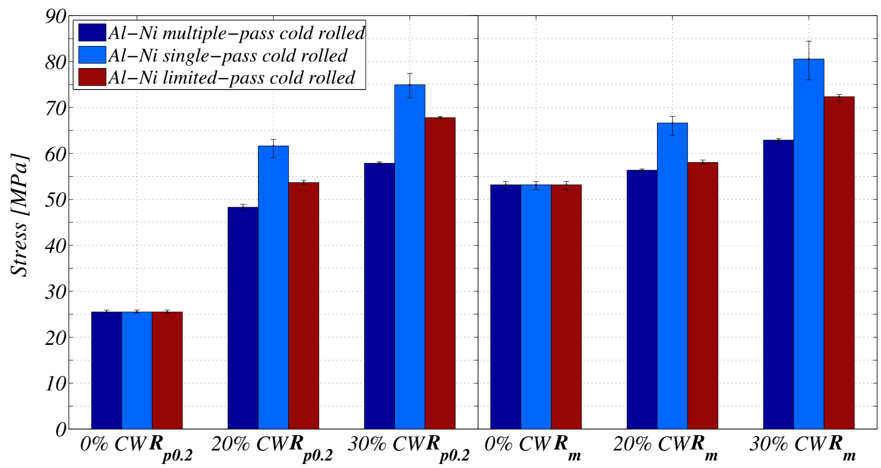

FIGURE 4. Tensile characteristics of the co-extruded conductor in three different cold-worked states. Properties are shown for the multiple pass, flat-rolled conductor, and for the single and limited pass, bi-directionally rolled conductor. Notice the decrease in tensile properties with amount of cold-roll passes.

In a previous study by the authors [5] the average mechanical values obtained were compared with results of earlier studies on the CMS and ATLAS solenoid conductor [6, 7, 19, 18]. Our values were found to be slightly lower than the gross of measurements conducted on the Al-0.1%Ni stabilizer material in the development of the CMS and ATLAS solenoid conductor. This observation lead us to the cautious conclusion that increased cross-sections result in decreased work-hardening effects. In the current study, however, significantly higher mechanical values are obtained for the same co-extruded Al-0.1wt%Ni stabilizer. The results are still fairly low in comparison with the earlier studies on the ATLAS solenoid conductor, supporting this effect of increased cross-section, however an additional effect has been observed. In Fig. 4 presented are the $R_{p0.2}$ and R_m values from our previous study, as well as the ones obtained here. A distinct increase is visible from the previous manually-driven flat rolling by two rollers, to the current bi-directional rolling by four rollers, done on a production scale. In the first case a certain reduction was achieved by multiple passes, whereas in the second case the same reduction was achieved in a single pass. The third bar represents the cold-rolled conductor on a production scale, where a certain reduction was achieved in a limited amount of passes by steps of

10% of the initial cross-sectional area. Apparent is the decrease in mechanical properties with the amount of passes. The explanation may relate to the phenomenon of age-softening, found more frequently in high-purity aluminum, aluminum alloys with a high-purity aluminum matrix, and other light alloys [20]. Age-softening is the occurrence of a loss of strength and hardness which takes place at room temperature due to the spontaneous decrease of residual stress in the strain-hardened structure. Age-softening in-between passes could result in the decreased effect of cross-section reduction on the mechanical properties of the Al-Ni stabilizer material.

Fig. 5 displays the relationship between $R_{p0.2}$ and RRR for the multiple pass, single pass and limited pass cold-rolled cases. The data indicate that although the lines are shifted with respect to each other, a similar relationship exists between RRR and $R_{p0.2}$ at room temperature for the various work-hardening processes.

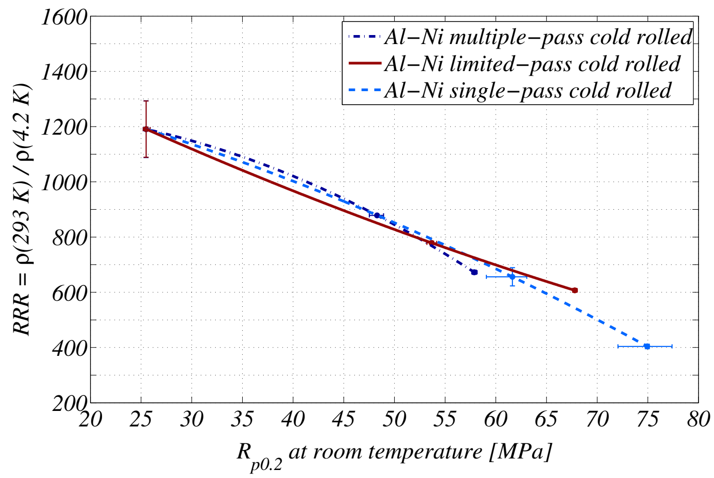

FIGURE 5. RRR plotted as function of $R_{p0.2}$ for the various cold-worked states, for the different work-hardening processes. It is seen that although the lines are shifted with respect to eachother, the relationship between RRR and $R_{p0.2}$ remains equal for all work-hardening processes.

When we compare the delamination of the 20% cold-rolled conductor, reduced in a single pass (Fig. 2c1), with the delamination of the 20% cold-rolled conductor, reduced in two passes (Fig. 2e), we observe a distinct difference. In addition to a decrease in mechanical properties, a decrease in delamination intensity is apparent. The relieve of internal stress in between cold-work passes appears to have an influence on the bonding quality of the conductor. Furthermore, from Fig. 2e, which displays an ultrasound image for a conductor which was homogeneously reduced by a single pass, by an equal reduction percentage in width as well as in thickness to reach the same cross-section reduction, we conclude a less severe delamination. The delamination in this case is concentrated at the edges of the Rutherford cable, most likely due to large stabilizer material movement in this area when homogeneously reduced.

CONCLUSION

An extensive qualification of post-extrusion thermo-mechanical treatments is carried out on a record size co-extruded Al-0.1wt%Ni stabilized superconductor. The expected increase in $R_{p0.2}$ with dilute Ni alloying is confirmed, and the enhancement of the mechanical properties at 4.2 K is promising for the low temperature application.

The comparison of various work-hardening processes, flat-roll multiple pass, bi-directional single pass and bi-directional limited pass, demonstrated a distinct increase of mechanical properties with a decrease in number of cold-roll passes. Supposedly there exists a process of recovery of dislocation pinning points by age softening in between cold-work passes for these very dilute aluminum alloys. We do notice a nearly linear relationship between RRR and $R_{p0.2}$, independent on the number of passes. From the above we can conclude that the mechanical and resistivity characteristics of Al-0.1wt%Ni are not only subject to the amount of work-hardening, but also to certain parameters of the work-hardening process.

The hardness values for the various thermally treated, 20% and 30% cold-reduced specimen, show no indication of precipitation hardening due to artificial aging. A recovery of the earlier applied work-hardening exists between a temperature of 450 and 530 K for the 20% and 30% reduced conductor subjected to a thermal treatment with a duration of 15 h. This implicates that the coil resin curing temperature, when applied for ∼15 h, should not exceed 450 K for the 30% cold-rolled conducter, and 470 K for the 20% cold-rolled conductor.

The results obtained by ultrasonic and radiographic measurements of the quality of bonding give rise to concern. Measurements conducted on conductor short samples cold-rolled to a cross-section reduction of more than 20% show clear indications of both delamination of the stabilizer material from the Rutherford cable, and cable damage. Ultrasonic images of various 20% cold-reduced conductor sections show evidence of a distinct relation between bonding quality and reduction procedure. Reduction in several passes is favorable for the bonding quality over the reduction in a single pass. Moreover, homogeneously compressing to a 20% reduced state results in a far lower extent of delamination, which is located differently. X-ray radiography showed some minor indications of strand damage, revealing that this high-force bi-directional rolling on large size co-extruded Al-0.1wt%Ni superconductors can only be performed without introducing defects up to a cross-section reduction of 15% (depending on conductor aspect ratio).

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