Paper CS-5 Presented at ICMC-CEC Albuquerque, Thursday July 15th 1993
Published in Advances in Cryogenic Engineering, vol. 40, pp. 725-732, 1994
This version contains corrections to typographic errors in Table III.

RESTRICTED NOVEL HEAT TREATMENTS FOR OBTAINING HIGH J_C IN Nb-46.5 WT.%Ti: II. PRESTRAIN DEPENDENCE

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ABSTRACT

In an earlier study, minimal initial heat treatments (3hr/300C) after a large cold work drawing strain (ε_p=6.37 and 7) were found to be very successful in providing nucleation sites for triple-point α-Ti precipitation during a final full heat treatment (80hr/420C). The minimal heat treatment schedules were repeated and expanded upon in this study but with a cold work pre-strain of only 5. The lower pre-strain reduced the effectiveness of the minimal heat treatment schedules resulting, typically, in a 25 % reduction in precipitate volume and in J_c. The J_c and volume % α-Ti values, however, remained remarkably high considering the minimal level of heat treatment and over 2800Amm^{-2} was achieved at 5T, 4.2K with only one conventional heat treatment (160hr/420C).

INTRODUCTION

The primary aim of the first series of experiments[1] was to maximize the efficiency of the multiple heat treatment and strain cycles used to produce high quantities of α-Ti and high critical current densities in Nb-46.5wt.%Ti. It was found that high critical current densities could be obtained within the restricted heat treatment strain space (ε_{HT} = 3.4) of that study by the introduction of additional heat treatments made possible by reducing the strain interval between heat treatments from the conventional $\Delta\varepsilon_{HT}$=1.15 to $\Delta\varepsilon_{HT}$=0.69. In this process, minimal (3hr/300C) intermediate heat treatments proved to be remarkably effective, provided that a long, higher temperature (eg. 420C) final heat treatment was applied. The low temperature heat treatments also produced a finer grain size prior to the final conventional heat treatment (although this refinement was lost in the final heat treatment). The most remarkable observation was the strong linear relationship between increasing volume % α-Ti and increasing J_c at both measured fields (5T and 8T, 4.2K). Subsequently this relationship was also found for Nb-50wt.%Ti.[2]

Following the success of this earlier experiment the matrix of heat treatments used for the original material was extended. In addition, the effectiveness of the low temperature

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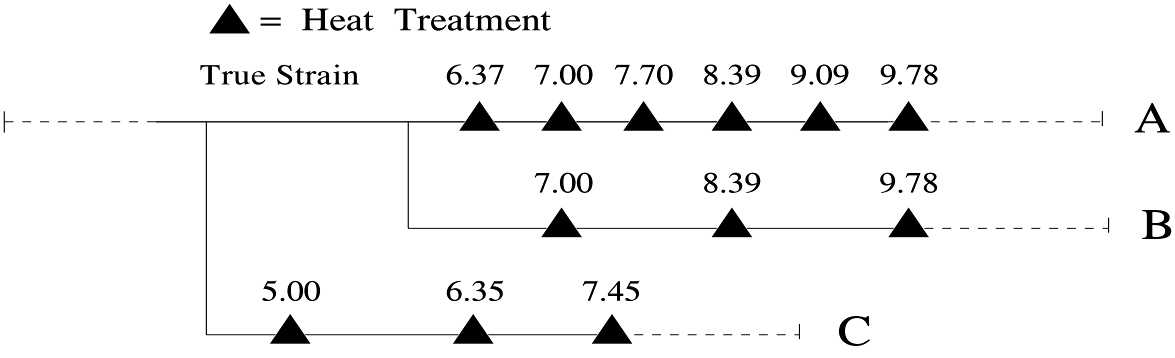

Figure 1 Schematic illustration of the heat treatment schedules in terms of cold drawn strain from final recrystallization anneal.

short duration heat treatment was examined in material where the “prestrain”, ε_p (extrusion and cold drawing strain prior to the first heat treatment) was restricted to the more normal 5, as compared to the 7 of the earlier experiment.

EXPERIMENTAL DESIGN

The initial alloy stock was a high homogeneity Nb-46.5wt.%Ti ingot supplied by Teledyne Wah Chang Albany (TWCA). The measured inhomogeneity was small, being ±0.5wt.% Ti over a 1mm scale region and a ±1wt.%Ti inhomogeneity over a 6mm scale for 146mm diameter billet after final recrystallization anneal (ε_t = 0).[1] The Nb-Ti monofilaments were wrapped in Nb diffusion barrier and clad in OFHC Cu at TWCA. The conductors were all monofilaments. They were cold drawn at the University of Wisconsin.

Optimizing the final Heat Treatment (Heat Treatment Series A)

In monofilaments designated UW230A, UW235A, UW240A, and UW245A, a six heat treatment sequence was applied, following the form of the first Novel Heat Treatment. The first five heat treatments were the same for each of the conductors (3 × 6hrs/375C + 20hrs/405C + 40hrs/405C). The final (6th) heat treatment was of varying time and duration. The final heat treatments were: 80hrs/420C (230A), 40hrs/420C (235A), 80hrs/405C (240A) and 80hrs/375C (245A). A schematic illustration of the heat treatment schedules is given in Figure 1. Heat treatments in schedule B and C will be described in the following sections. The cold work drawing strain prior to the first heat treatment was 6.37, and the

Table 1 . Heat Treatment Schedules for the 6 Heat Treatment “A” Series.

True Strain	Diam. mm	Heat Treatment Schedule, hr/C								
		210A	220A	230A	235A	240A	245A	250A	260A	270A
6.37	7.11	6/405	3/405	6/375	6/375	6/375	6/375	3/300	3/300	3/300
7.00	5.18	6/405	3/405	6/375	6/375	6/375	6/375	3/300	3/300	3/300
7.70	3.66	6/405	3/405	6/375	6/375	6/375	6/375	3/300	3/300	3/300
8.39	2.59	20/420	20/420	20/405	20/405	20/405	20/405	3/300	3/300	20/420
9.09	1.83	40/420	40/420	40/405	40/405	40/405	40/405	3/300	3/300	40/420
9.78	1.30	80/420	80/420	80/420	40/420	80/405	80/375	3/300	80/420	80/420
Peak J_c A/mm^2 5T, 4.2K		3223	3382	Results are given in this study.				2079	2973	3278

inter-heat treatment strain was 0.682 (3 standard die passes). The heat treatments are summarized in Table 1

Low Prestrain Restricted Heat Treatment Series (Series C)

Monofilament of the same stock was given an initial heat treatment at TWCA of 3hr/300C at a prestrain of 5 (the first series received their first heat treatments at a strain of 6.37). A second heat treatment of 3hr/300C was given at a prestrain of 6.35 followed by a final heat treatment at a prestrain of 7.45. The final heat treatments are listed in Table 2. Thus only three heat treatments were applied to the conductors, the initial two being very restricted in time and temperature (from earlier studies we would expect that these would only produce a grain boundary film).

Characterization

Critical Current Measurements. Critical current, I_c, measurements were performed on 0.6m lengths of wire which were wound on barrels mounted coaxially inside the bore of a 12T solenoid. The critical current density was determined at an overall wire resistivity of 10^{-14} Ωm (≈ 10µVm^{-1}). The critical current densities were calculated from the I_c values using Cu:NbTi ratios obtained by the etch and weigh technique.

Microstructural Characterization. The microstructural characterization was performed by Transmission Electron Microscopy, TEM.[3] The precipitates were analyzed for volume fraction and for the precipitate dimensions in transverse cross-section at final heat treatment size after heat treatment.

RESULTS

Final heat treatment Optimization Results

The critical current density versus strain behavior for the monofilaments is shown in Figure 2. The 80hr/420C final heat treatment produced the highest J_c values (3640 Amm^{-2} at 5T, 4.2K, 2200 Amm^{-2} at 7T, 4.2K and 1450 Amm^{-2} at 8T, 4.2K). Reducing the temperature of heat treatment, however, (80hrs/375C) produced higher J_cs at final strains below 4.6. For instance monofilament UW245A peaked at a final strain of 4.6 at 5T (3470 Amm^{-2}) and at 5.1 for 7 and 8T (2130 and 1410 Amm^{-2} respectively). The 80hrs/375C heat treatment also produced a broader peak in the J_c versus strain plot.

Low Prestrain Restricted Heat Treatments

A compilation of the 5T and 8T critical current results from this series is given in Table 2. This series yielded a wide range J_c values from ~600Amm^{-2} (5T,4.2K) for the 3hr/300C only series to over 2800Amm^{-2} (5T,4.2K) for a final heat treatment of 160hr/420C (exceeding SSC J_c specification)! Comparing the high prestrain three heat treatment series (B) with this material we find a 20% lower J_c at 5T (UW260B compared with UW255C). As in the previous heat treatment series the final strain for peak J_c increased with the final heat treatment temperature. Relatively high strain to peak J_c values were required for all this series (a peak in J_c versus strain had not been reached at 5.78 - our maximum strain the 435C and 160hr/420C wires). The strain to peak J_c ranged from a strain of 5 for 375C to more than 5.8 for 420C and 435C. This compares with a strain of 4.6 at 375C and of 5 for 420C in the first (high pre-strain) series. However, the total strains were lower, due to the earlier and more closely spaced heat treatments. Peak J_c values increased with both

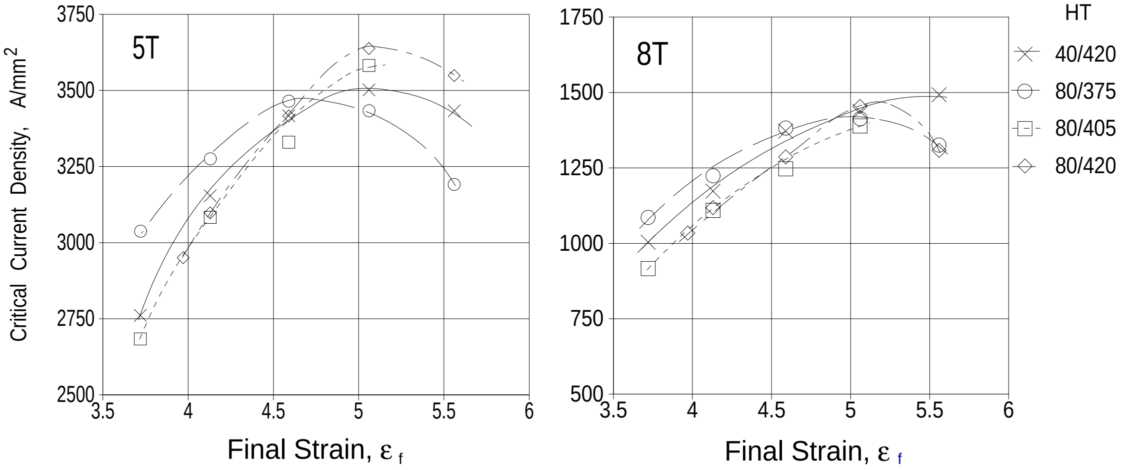

Figure 2. Critical current density versus final drawing strain for monofilaments from series A given 3 x 6hr/375C, followed by 20hr/405C, 40hr/405C heat treatments with a final heat treatment as given in the key.

time and temperature. Increasing the temperature of heat treatment from 375C to 405C increased the J_c at 5T by about 10% (Figure 3) with smaller but significant gains through to 435C (typically 0-5% for every 15°C). An increase in J_c of 10-17% could be obtained by increasing the heat treatment time from 80-160hrs. The 3hr/300C low prestrain conductors had J_c values close to those of non-heat treated material with no peak in J_c with strain. This contrasts with the high prestrain equivalent conductor where thee was a distinct peak at a final strain of 4.13 (J_c = 940Amm^{-2}, 5T, 4.2K).

Table 2 Critical current data at 5T and 8T, 4.2K for ultra-restricted heat treatment series.

Conductor ID	Final Heat Treatment, hr/C	J_c, 5T, 4.2K, Amm^{-2} at Final Strain					J_c, 8T, 4.2K, Amm^{-2} at Final Strain				
		4.6	4.84	5.11	5.32	5.78	4.6	4.84	5.11	5.32	5.78
UW205C	3/300	619	572	525	596	540	441	414	403	436	409
UW210C	20/375	1760	2004	1798	1754		892	1033	946	919	
UW215C	40/375	2032	2050		2069	1998	981	999		1047	1052
UW220C	60/375	2123	2180	2190	2146	2081	996	1033	1068	1064	1064
UW225C	80/375	2207	2151	2208	2148	2093	1018	1010	1056	1044	1048
UW230C	20/405	1963	1941	1968	1970	1895	949	953	976	990	981
UW270C	40/405	2196	2244	2281	2267	2223	998	1046	1052	1079	1121
UW235C	60/405	2283	2340			2330	1018	1058	1081		1132
UW240C	80/405	2184*	2309		2425	2391	960*	1047		1143	1164
UW275C	160/405	2445	2566	2627	2646	2649	1016	1108	1143	1179	1247
UW245C	40/420	2182	2317	2291	2272	2297	983	1067	1082	1081	1149
UW250C	60/420	2266	2244	2423	2518	2323	1004	1020	1125	1158	1128
UW255C	80/420	2251	2328	2384	2414	2408	994	1012	1058	1105	1144
UW280C	160/420	2415	2516	2629	2752	2837	979	1050	1116	1193	1294
UW260C	40/435	2131	2200	2179	2216	2237	927	960	999	1032	1095
UW265C	80/435	2226	2240	2336	2458	2519	943	968	1033	1106	1171
B Series[1]	Final Heat Treatment, hr/C	J_c, 5T, 4.2K, Amm^{-2} at Final Strain					J_c, 8T, 4.2K, Amm^{-2} at Final Strain				
		3.72	4.13	4.59	5.06	5.56	3.72	4.13	4.59	5.06	5.56
UW250B	3/300	713	942	596	651	564	484	480	442	429	415
UW260B	80/420	2051	2497	2735	2922	3044	730	895	1017	1156	1280

* = at a final strain of 4.39

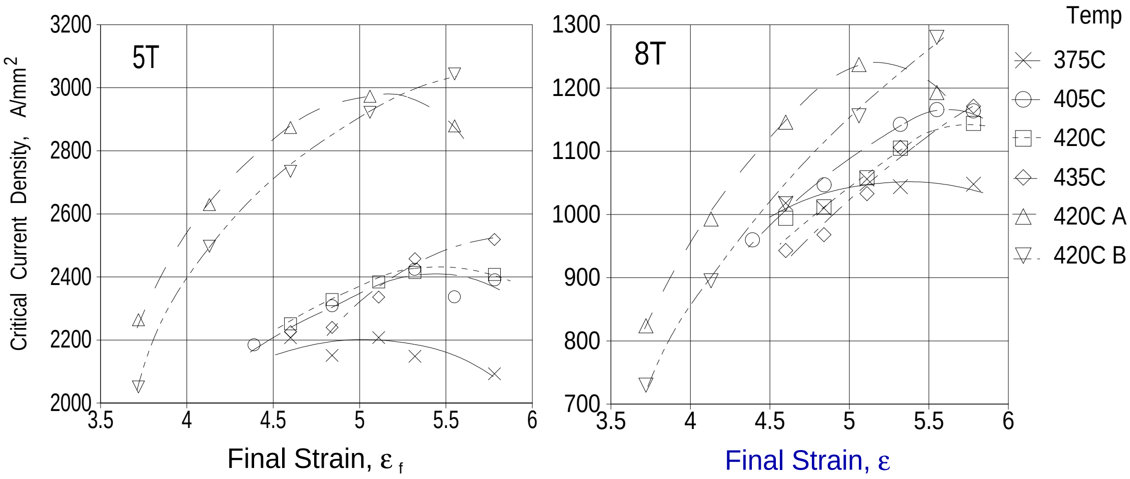

Figure 3 J_c versus final strain for low prestrain restricted series C monofilaments with 80hr final heat treatments. This data is compared with the significantly higher critical current densities obtained in the equivalent high pre-strain series: "A" (5 x 3hr/300C + 80hr/420C, $\Delta\varepsilon_{HT}$=0.69) and "B" (2 x 3hr/300C + 80hr/420C, $\Delta\varepsilon_{HT}$=1.38).

Quantification of the α-Ti Precipitation

The results of the microstructural analyses are given in Table 3. The lower prestrain in the low prestrain restricted series is clearly producing a lower volume of precipitate. These results can be incorporated into the peak J_c versus volume percent α-Ti data of the first data (Figure 4) along with the non-heat treated data of Warnes and Larbalestier.[4] Despite the difference in the prestrain and inter-heat treatment strains, all the data from these series fits closely to the linear relationship established in the first paper. The precipitate size distributions are quite different for the low prestrain restricted series, as compared to the high pre-strain material. This difference is illustrated in the histogram in Figure 5. As the wires in the low prestrain restricted series have a lower overall strain at the final heat treatment size than series "A" and "B", an additional set of data has been generated for Figure 5 in which the precipitate diameters have been reduced as if uniformly

Table 3. Quantitative measurements of the microstructure immediately after final heat treatment

Wire ID	Heat Treatment	Peak J_c		Microstructural Quantification			
		5T (ε_f)	8T (ε_f)	α-Ti	Average α-Ti Cross-Sectional Area		$d^*_{\alpha\text{-Ti}}$, nm
		Amm^{-2}		Volume %	Mean, nm^2	Median, nm^2	
UW230A	3 x 6hr/375 + 20hr/405 + 40hr/405 + 80hr/420C	3638 (5.1)	1454 (5.1)	22.6	21537	10647	166
UW240A	3 x 6hr/375 + 20hr/405 + 40hr/405 + 80hr/405C	3582 (5.1)	1389 (5.1)	23.6	25262	10755	179
UW245C	2 x 3hr/300C + 40hr/420C	2317 (4.8)	1149 (5.8)	10.3	11665	2467	122
UW255C	2 x 3hr/300C + 80hr/420C	2414 (5.3)	1144 (5.8)	16.7	17077	3830	147
UW265C	2 x 3hr/300C + 40hr/420C	2519 (5.8)	1171 (5.8)	13.9	13347	2958	130

d* = mean diameter of α-Ti precipitate calculated from mean cross-sectional area assuming circular cross-section drawn down with the wire to the final heat treatment size for series A and B. Whichever histogram for UW255C is chosen, the precipitate distribution is non-Gaussian for the low prestrain material with an asymmetrical peak shift to low precipitate size. This contrasts with the more uniform distribution for the high pre-strain material. Although the 3hr/300C heat treatments have no impact on J_c themselves for the low prestrain material they still increase the volume % of alpha-Ti produced by the 80hr/420C heat treatment (16.7 volume % compared with ~11% produced by a single 80hr/420C heat treatment[5]).

CONCLUSIONS

Final Heat Treatment Optimization

1. Although the 80hrs/375C process produced a lower peak J_c than 80hrs at 405C or 420C, presumably due to a lesser amount of precipitate, it produced higher J_c values at low final strains, possibly due to reduced coarsening of the microstructure during the final heat treatment.

2. Our studies of intermetallic formation and sausaging suggest that lower temperature heat treatments are beneficial if thin barriers are used. Therefore long low temperature final heat treatments could be useful in both reducing final strain for optimization and decreasing the amount of intermetallic.

Low Prestrain Restricted Heat Treatments

1 TEM on the first series (UW2xxB) indicated that the 3hrs/300C heat treatments acted primarily as precipitate nucleators, while retaining a refined scale microstructure. At the lower prestrain these heat treatments still increased the volume % of α-Ti precipitate compared to a single heat treatment but there was significantly less of an enhancement than for the high prestrain conductors. The 3hr/300C heat treatments alone appeared to have no impact on J_c for the low prestrain conductors compared with non-heat treated conductor.

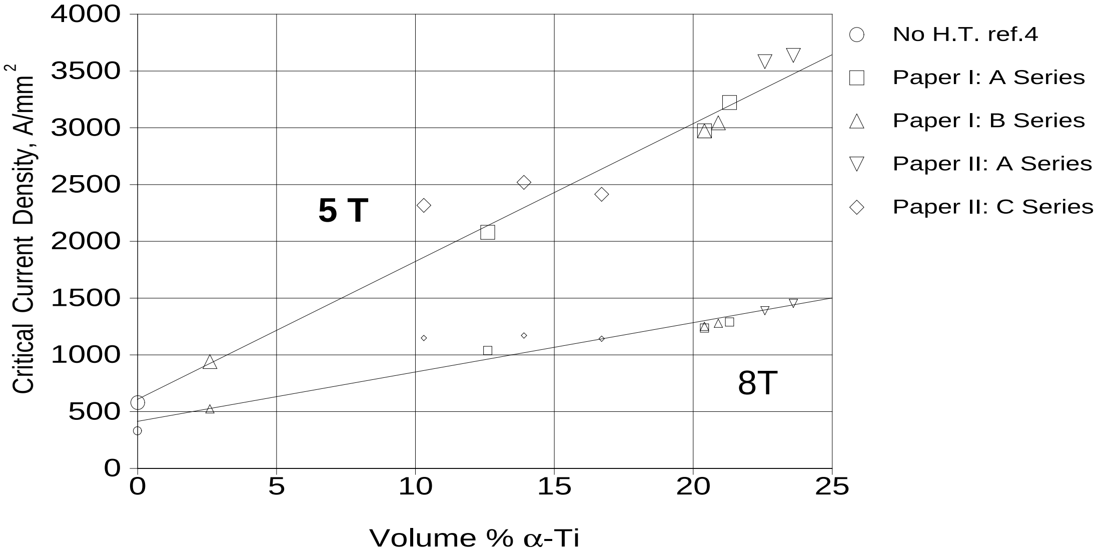

Figure 4 Peak J_c versus cross-section % α-Ti for papers I and II, series A,B and C. The no-heat treatment results are from Warnes and Larbalestier.[4]

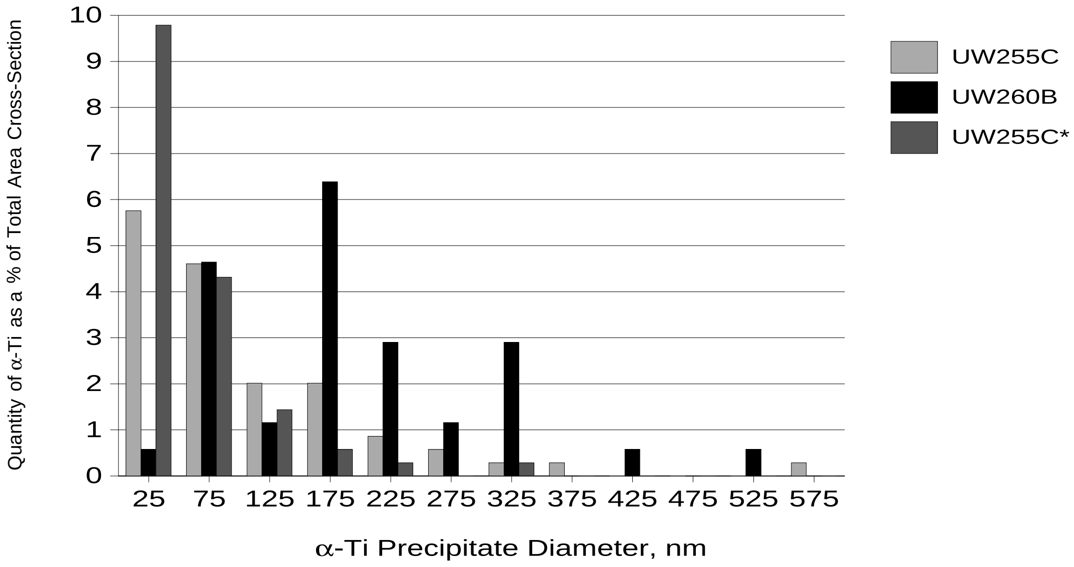

Figure 5 Precipitate diameter (calculated from the cross-sectional area assuming a circular cross-section) distribution for 255C and 260B at final heat treatment size, equivalent conductors of the low and high prestrain three heat treatment series respectively (2 x 3hr/300C + 80hr/420C). * = estimated diameters for 255C if the precipitates had been uniformly drawn to the same wire diameter as 260B, calculated by applying the required wire diameter reduction to the calculated precipitate diameter

2 The total strain to peak J_c in the low prestrain series was 1.8 less than the first series, while the final strain was approximately 0.5 more. Thus the prestrain difference had a relatively small impact on final strain to peak J_c compared with the influence of final heat treatment conditions.

Quantification of α-Ti Precipitate after Final Heat Treatment

1 There is a good fit to the linear volume % α-Ti versus peak J_c relationship established in the earlier study[1] for all conductors in these two studies regardless of prestrain or heat treatment condition.

2 Reducing prestrain reduces precipitate volume, as well as size uniformity.

3 The 3hr/300C heat treatments increased the volume of precipitate produced by the final heat treatment. The increase in the level of precipitate was not as great for the low prestrain material as for the high prestrain B series indicating the strong strain sensitivity of this effect.

ACKNOWLEDGEMENTS

The work was supported by the Department of Energy, Division of High Energy Physics under Grant #DE-AC02-82ER-4007. The initial heat treatment at low strain was performed by Phil O'Larey at Teledyne Wah Chang Albany. Additional wire fabrication and I_c measurements were performed by Alex Squitieri, Bill Starch, And Bob Remsbottom (now at SSCL).

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