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EFFECTS OF CYCLIC STRAINS ON TRANSPORT PROPERTIES
OF A SUPERCONDUCTING COMPOSITE:
PHASE I, DEGRADATION OF ELECTRICAL CONDUCTIVITY
IN COPPER AT 4.2 K

by

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ABSTRACT

The effects of cyclic strains at 4.2 K on the electrical resistivity of copper have been investigated as Phase I of a program to determine the overall effects on monolithic superconducting composites. The major part of the work consisted of cycling of C101 oxygen-free electronic (OFE) copper samples in the constant-strain mode with strain amplitudes of 0.05-0.4%. The resistivity increase was very significant when well-annealed samples were cycled at strain amplitudes greater than 0.15%. For example, after 2000 cycles at 0.20% strain amplitude, the resistivity produced by both point defects and dislocations is nearly the same as the transverse magneto-resistance at 80 kG. The primary factor in determining the induced resistivity in commercially available copper stock is the recrystallization texture. The least resistivity was produced in heavily rolled stock with sharp $\langle 111 \rangle$ fiber textures. About 55% of the resistivity produced by 200 or more cycles anneals out after 2 days at 300 K. A comparison of the present data with literature data for unidirectional deformation indicates that, for comparable accumulated strain, unidirectional deformation is much more effective in producing point defects.

I. INTRODUCTION

This work is a direct application to the design of large superconducting magnets that are subject to several different modes of cyclic strain during assembly and normal operations.

The specific purpose of this phase of the program, and of this final report on Phase I, was to evaluate the total increase in the bulk resistivity, ρ_0 , of copper at 4.2 K after well-defined cyclic-strain modes. The most important of these modes will be hoop stress, a variation created during energizing and deenergizing of the magnet. To relate the tension and compression components of strain to $\Delta\rho_0$, the change in ρ_0 , an apparatus was built to produce push-pull (pure tension-compression) cycling of relatively large

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copper samples of different purity and fabrication history. The ρ_0 after a given number of cycles was measured in the most direct manner by the four-point-probe, voltage-drop technique. The results of the measurements were highly gratifying in that the values of $\Delta\rho_0$ measured for a given set of conditions were reproducible to within 5%. As a consequence, the data have permitted us to determine the relative importance of several factors that contribute to the rate of change of ρ_0 with the number of constant-strain cycles and to make some reliable quantitative predictions of such rates of change as a function of the following parameters:

1. Grade of copper, as defined by standard ASTM and CDA designations.
2. Fabrication and annealing history of the material.
3. Amplitude of strain per cycle.
4. Frequency of strain cycle.

For practical reasons, we have limited the range of variation in each parameter to those areas most relevant to conditions expected in large superconducting magnets such as those proposed for the Fusion Energy Program. However, the experiments were designed to provide sufficient understanding of the source of the $\Delta\rho_0$ so that extrapolation from the data to more extreme conditions would be based on valid models. The scope of the work accomplished under this program was sufficient to (1) provide a reliable basis for specifying the optimum purity and heat treatment of the copper to be purchased and (2) obtain reliable estimates of the increase in ρ_0 during cyclic straining of any particular copper during magnet operation.

II. BACKGROUND

A. Relation of Cyclic Stress Effects to Other Factors Contributing to the Resistivity of Copper

1. Definition of ρ_0

The ultimate purpose of the present research is to enable the designer of large high-field, copper-stabilized superconducting magnets to evaluate the total resistivity, ρ_0 , for the stabilizer at operating temperatures and magnetic fields. The parameter ρ_0 represents the residual or temperature-independent resistivity at $T < 10$ K. At these very low temperatures, ρ_0 is essentially determined by the additive contributions to electron scattering,

$$\rho_0 = \rho_i + \rho_m + \rho_d, \quad (1)$$

where ρ_i is the impurity contribution, ρ_m is the magnetoresistance, and ρ_d is the contribution from the crystalline imperfections, dislocations, and point defects.

According to Matthiessen's rule, ρ_i and ρ_d are independent of temperature and, at zero magnetic field and $T < 10$ K, ρ_0 in a well-annealed copper is a measure of the impurity content. Typical values of ρ_i for copper range from $0.5 \text{ n}\Omega\cdot\text{cm}$ for research-grade 99.999%-pure stock (5N) to $\sim 10 \pm 3 \text{ n}\Omega\cdot\text{cm}$ for the commercial-grade C101, oxygen-free electronic (OFE) copper. (A more detailed description of commercial grades appears below in Sec. II.B.) At higher temperatures, i.e., $T > 15$ K, the phonon contribution, $\rho(T)$, begins to appear and rapidly increases with temperature to overwhelm ρ_0 . For copper at 300 K, for example, $\rho(T) \approx 1.70 \pm 0.02 \text{ }\mu\Omega\cdot\text{cm}$, regardless of purity or defect concentration, and the ratio ρ_{300}/ρ_0 is a useful measure of the level of impurities and defects in copper and other metals. This parameter is generally referred to as the RRR, i.e., residual resistivity ratio.

In line with the values for ρ_0 noted above the RRR for recrystallized copper varies from ~ 3400 for a well-annealed high-purity 5N grade to $\sim 200 \pm 25$ for well-annealed OFE grade C101. An RRR of 225 ± 10 is typical of the OFE samples tested in this study and also reported in recent investigations.^{2,3} Similar or even higher RRR values are often found in the electrolytic-tough-pitch (ETP) grades, designated C110, and also in the free-machining grades C147 and C145, with added sulfur or tellurium. However, the RRR values for these lower grades of copper are not generally uniform in a given billet and can therefore vary significantly from the 225 value.

2. Transverse Magnetoresistance

The increase in ρ_0 in a transverse magnetic field, H , is a significant factor in considering cryogenic stabilization of a large superconducting magnet. The magnitude of ρ_m can be reasonably well predicted from Kohler's rule,⁴ where $(\rho_m/\rho_0)(H=0) \sim H(\text{RRR})_0$ and $(\text{RRR})_0$ is the value of RRR at $H = 0$. The data for several grades of copper with wide variations in RRR are consistent with this rule.⁵ For fields with $H > 10$ kG, it has been noted⁵ that

$$(\rho_m/\rho_0)(H=0) = 0.23 \times 10^{-6}(\text{RRR})_0 H/\text{gauss}. \quad (2)$$

Since ρ_0 at $H = 0$ appears in the denominator of both sides of Eq. 2 and $\rho_{300} \approx 1.70 \text{ }\mu\Omega\cdot\text{cm}$ for any grade of copper, the approximate value of ρ_m can be obtained from

$$\rho_m = 0.23 \times 10^{-6}(1.70 \text{ }\mu\Omega\cdot\text{cm})H/\text{gauss}. \quad (3)$$

From Eq. 3, $\rho_m \approx 15 \text{ n}\Omega\cdot\text{cm}$ at 40 kG and $\sim 30 \text{ n}\Omega\cdot\text{cm}$ at 80 kG. For a large superconducting magnet producing 50-100 kG at the conductor, the value of ρ_0 for the commercial copper stabilizer during operation will exceed $22 \text{ n}\Omega\cdot\text{cm}$ at 50 kG and $47 \text{ n}\Omega\cdot\text{cm}$ at 100 kG (based on $\sim 7 \text{ n}\Omega\cdot\text{cm}$ for ρ_i).

3. Defect Resistivity

The defect resistivity, ρ_d , has two basic contributions: the dislocation contribution ρ_D and the point-defect contribution ρ_P . Although dislocations exist at the grain boundaries of an annealed polycrystal, the high densities of dislocations that affect ρ_0 are produced by plastic deformation at temperatures below the recrystallization temperature. For copper, the lowest temperatures for any degree of recrystallization are above ~ 423 K, so that dislocations introduced during plastic deformation at lower temperatures will remain in the structure as long as the temperature remains below ~ 423 K.

There is extensive information regarding the change in ρ_0 with deformation at room temperatures⁶ and the combination of these data with measurements of stored energy, electron microscopy, and macroscopic density show that the ρ_D can be reasonably well estimated from $\rho_D = 1.3 \times 10^{-19} \Omega \cdot \text{cm} / \text{unit dislocation density}$, with only a small dependence on temperature.⁶ The fact that heavy cold working at room temperatures generally produces an RRR of ~ 55 -60, or ρ_0 values of 28 - $30 \text{ n}\Omega \cdot \text{cm}$ can thus be understood as arising from dislocation densities of 1 - $3 \times 10^{11} / \text{cm}^2$. For C101 copper, this represents a contribution of $\sim 22 \text{ n}\Omega \cdot \text{cm}$ from ρ_D , or about the same as ρ_m at 50 kG . Annealing to 200°C or greater is then clearly advantageous in reducing ρ_0 .

The point defects and defect clusters that are responsible for a significant ρ_P can be produced by several sources, such as irradiation damage from neutrons⁷ or charged particles,⁸ quenching from a high temperature,⁹ or dislocation interactions during plastic deformation.¹⁰⁻¹³ In this program we are concerned only with the last of these sources. The primary difference in the impact of point defects versus ρ_D is that many of the point-defect clusters produced by plastic deformation generally anneal out at temperatures between 20 and 250 K . Thus, ρ_P is generally insignificant as a result of deformation at room temperatures or higher, but remains as a contribution to ρ_0 when the metal is deformed at lower temperatures and cooled to $T < 15 \text{ K}$.

The relative ease of motion for point defects is apparent from a number of basic studies of the formation and annealing kinetics of vacancies and interstitials in copper introduced by neutron¹⁴ and electron bombardment¹⁵ and for vacancies retained by quenching.⁹ It is clear from these data that close-pair vacancy-interstitial annihilation begins at about 15 K for copper and that there are three prominent annealing stages between 15 and 300 K . Although the proposed models for the different stages are not accurate in detail, the higher-temperature annihilation stages are associated with the mobility of single vacancies and vacancy clusters. In addition, the most recent data for copper indicate that $\rho_P = 2.0 \mu\Omega \cdot \text{cm}$ per $1 \text{ at. \% Frenkel pair}$.¹⁶

The most definitive data regarding defects produced by plastic deformation of copper at 4.2 K have emerged from the single-crystal studies of Blewitt et al.¹² These studies showed that (1) the ρ_d after deformation at 4.2 K is dependent on the square of resolved shear stress on the primary

glide system, (2) the shear stress to attain a given shear strain is significantly dependent on whether single- or multiple-slip systems are operating, and (3) no significant annealing, or decrease in ρ_d produced at 4.2 K, occurs at $T < 78$ K. Points 1 and 2 established the importance of the dislocation interactions, rather than strain alone, in determining the number of point defects. The third point established that the defects were primarily vacancies, since single interstitials should be mobile and thus cause ρ_d to decrease at $T < 78$ K.

Other studies¹⁶ indicate that the annealing of the vacancies occurs in two stages, the first at 170 K and the second at 240 K. The first is probably associated with clusters or rows of vacancies, whereas the 240 K stage corresponds to single vacancies.

Data obtained by W. B. Pearson¹³ (Fig. 1) show the significant effect of the temperature of unidirectional deformation of copper. Curve C shows that room temperature or ordinary cold working to 50-100% reduction in area (R.A.) will contribute about 25-30 $\text{n}\Omega\cdot\text{cm}$ to ρ_0 , whereas deformation to 57% R.A. at 4.2 or 78 K (Curve A) will give equivalent ρ_d values near 150 $\text{n}\Omega\cdot\text{cm}$. After aging at room temperature, about 55% of ρ_d is annealed out, as shown in Curve B, but the remaining resistivity is about 55 $\text{n}\Omega\cdot\text{cm}$ greater than that deformed at room temperature. This suggests that either the dislocation density after 57% deformation at 78 or 4.2 K is almost twice that obtained in room-temperature cold rolling or that a significant fraction of point defects remain as effective electron scatterers. The latter explanation

Fig. 1. Variation of RRR and ρ_0 with Percent Reduction in Cross-sectional Area for Unidirectional Cold-worked Copper Deformed under the Following Conditions: (O) 4.2 K in Tension, (●) Rolled at 77 K, (□) Ambient-temperature Tension and Aging, and (■) Ambient-temperature Rolling and Aging.¹³ (Δ) denotes aged at ~300 K after 4.2 K tension, and (▲) denotes aged at ~300 K after rolling at 77 K. Neg. No. MSD-64233.

is suggested by the observation by Blewitt et al.¹² of a strain-aging effect due to room-temperature annealing in single crystals. The effect is believed to be one of vacancies migrating to pin dislocations, but there is little evidence that will clearly identify the mechanism. The effect is emphasized here because it was very pronounced in the low-temperature cycling experiments described in this report.

B. Prior Work with Stress or Strain Cycling

Although some experiments have been carried out to determine $\Delta\rho$ for copper after deformation cycling at low temperatures, none have been carried out with conditions that can be directly related to constant-strain cycling at 4.2 K, with uniform deformation over a massive sample. The two most relevant investigations are (1) the constant-load cycling at 4.2 K by Johnson and Johnson,¹⁷ with resistivity determined from surface electrical impedance, and (2) the constant-strain torsion cycling and dc voltage measurements at 78 K by Polak.¹⁸ The former data for OFE copper were dominated by the work-hardening effect causing a decreasing strain per cycle and saturation in $\Delta\rho$ after less than 500 cycles.

The data showed, nevertheless, that the initial strains per cycle of 0.3% can contribute about 20 n Ω ·cm after 1000 constant-stress cycles of annealed OFE-grade copper. They also showed that about 60% of $\Delta\rho$ anneals out during room-temperature anneals and that this annealing is associated with an increased yield stress during subsequent 4.2 K cycling. The present data confirm the qualitative conclusions of Ref. 17 and are also in approximate agreement with the initial $\Delta\rho$ values obtained in the latter. Because of the changing strain per cycle in constant-stress experiments, the Ref. 17 data are not of sufficiently quantitative accuracy to be of value in design work.

The other paper of special interest is that of Polak,¹⁸ in which constant torsional strains were introduced to 1-mm-dia wire samples at 78 K. When the torsional strains are converted to equivalent tension-compression strains, the $\Delta\rho_d$ values are much larger than indicated by Ref. 17 constant-stress data. At ~0.30% tensile-compression strains, Polak's data have $\Delta\rho$ values after 1000 cycles of 60 n Ω ·cm, rather than the 20 n Ω ·cm of Ref. 17.

Another feature of the Polak data is that the shear stress, $\bar{\tau}$, to attain a given constant strain and $\Delta\rho$ reached saturation after a relatively few cycles. For 0.30% strain, both $\bar{\tau}$ and $\Delta\rho$ reached saturation after ~1000 cycles. In this respect, Polak's data are similar to those of Ref. 17 and suggest that the torsional strains were completely elastic after 1000 cycles. The conclusions from a comparison of the two sets of data indicate that torsional tests produce considerably more work-hardening per cycle than in push-pull compression tests. It also is clear that testing with well-controlled constant strains per cycle was needed to predict the $\Delta\rho$ for push-pull cycling.

III. EXPERIMENTAL PROCEDURES

A. Tension-Compression Apparatus

The stress cycling was produced by an Instron mechanical testing machine, Model TTDCM-L with crosshead speeds of 0.03-5.0 cm/min and a load capability to 10,000 kg. Figure 2 is a schematic representation of the assembly, and Fig. 3 is a photograph of the apparatus before immersion in the cryostat. The massive copper test samples were coupled to the load cell in the fixed beam of the machine, and the lower grip end was attached to an outer sleeve that moved up or down with the moving beam. Lateral movement of the sample was restricted by a flexible diaphragm through which the load-cell coupling rod moved in the push-pull mode. The assembly was immersed in liquid helium contained in a cryostat fixed to a flange on the moving beam.

Figure 4 is a cross-sectional scale-drawing view of the fatigue assembly showing complete design details. A typical sample with extensometer and electrical current leads in position is shown in Fig. 5.

B. Strain-gauge and Electrical-resistivity Measurements

The specimens were attached to the fatigue assembly by means of split stainless steel grips shown in Fig. 6. One set of grips was mounted on each end of the specimen, and the specimen-grip nut, shown in Fig. 7, fitted firmly over the grips to bolt the specimen in place.

Figure 8 shows the extensometer assembly for measuring the strain and the voltage across the gauge length of the specimen. The two strain gauges that monitored strain amplitude and the voltage probes used in

Fig. 2. Schematic Drawing of Assembled Cycling Apparatus, as Attached to Mechanical Testing Machine and Immersed in Liquid-helium Cryostat. Neg. No. MSD-59777.

Fig. 4

Design Details for Sample Grip Assembly. Inner-tube push rod extends up to load cell on stationary beam of machine. Outer tube extends to moving beam of machine and thus transfers vertical displacement to sample. Neg. No. MSD-59778.

Fig. 3

Cycling Apparatus before Installation of Cryostat. Neg. No. MSD-59745.

Fig. 5

Sample in Assembly with Extensometer and Voltage Probes Attached. Neg. No. MSD-59743.

Fig. 6

Split Steel Collar for Rigid Grip on Sample Ends. Neg. No. MSD-59749.

Fig. 7

Steel Collar Nut for Rigid Coupling of Sample Ends to Push Rod and Moving Beam. Neg. No. MSD-59746.

Fig. 8

Closeup View of Extensometer Assembly. Four SR-4 strain gauges were cemented to aluminum spring clamps. Neg. No. MSD-59775.

measuring the changes in electrical resistivity were attached to two U-shaped extensometer spring clamps mounted directly on the specimen. Brass knife edges served as voltage probes and were attached to, but electrically isolated from, the extensometer clamps. The distance between the voltage probes was 2 cm.

Four strain-gauge resistors (SR-4 FNB-12-35) were glued to the closed end of the U clamps, with one resistor on each face of both clamps. A resistance bridge constructed from the four strain-gauge resistors determined the strain amplitude.

Figure 9 is a block diagram of the instrumentation used to measure electrical resistivity and stress-strain characteristics of the specimen during cycling. The electrical resistivity of the specimen was obtained by means of the standard four-probe dc technique. The current from the dc power supply was monitored by a digital-voltmeter probe across a $10^{-3}\text{-}\Omega$ standard resistance, and the voltage across the 2-cm gauge length was determined from a Keithley 149 nanovoltmeter/amplifier. The recorded voltage was the average of two readings differing in the current direction. The reproducibility of a measurement was generally within $\pm 5\text{ nV}$ for a 10-A current. With the 0.635-cm sample gauge diameter and 2-cm gauge length, a $\Delta\rho_0$ of $0.2\text{ n}\Omega\cdot\text{cm}$ could readily be detected.

Fig. 9. Block Diagram of Electrical Circuits Used to Monitor Stress, Strain, and Voltage across Gauge Length of Copper Sample. Neg. No. MSD-59776.

C. Description of Copper Samples

The purchase of copper for high electrical conductivity at temperatures below 20 K requires a careful understanding of standard terminology in the copper industry. This care is particularly important among the commercial grades that may be considered for current sharing in a superconducting

composite, where resistivity ratios of 175-250 are expected in the annealed state. It appears to be quite important to follow the designations of the Copper Development Association and the Unified Numbering System for Metals and Alloys (UNS) adopted jointly by ASTM and SAE.¹⁹ The primary grade considered in this study is that designated C101, with OFE (oxygen-free electronic) designation. Some tests were also carried out with a free-machining grade designated C147, containing between 0.2-0.5% sulfur, as well as with a very high-purity research grade containing 99.999% copper (designated in this report as 5N).

One of the principal results of this study is that the fabrication history of the material, as it affects the preferred orientation of the grains, is an important factor in determining $\Delta\rho_0$ with strain cycling. Consequently, the description of the different groups of samples listed in Table I contains the known history before machining of the samples from the raw stock. One group of C101 (Group 2 of Table I) samples were machined directly from the rod as received from the vendor as a 1-in. (2.54-cm)-dia rod. This rod was presumably "hot-rolled" from the original billet, but metallographic examination indicated that the final reduction to 1-in. (2.54-cm) diameter was a cold-worked operation. This group consisted of four samples. Another rod from the same vendor, also 1 in. (2.54 cm) in diameter, was further cold-rolled to 5/8-in. (1.6-cm) diameter prior to machining, and yielded eight samples making up Group 3 of Table I. The stock for Group 4 was similar in history to that for Group 3, but was obtained from a different source.

TABLE I. History of Copper Samples for Cyclic-strain Studies

Sample Group	Source	Grade or UNS Number	Fabrication before Machining	Anneal after Machining		RRR
				Temp, °C	Time, h	
1	ASARCO	5N	Annealed as received, 3/4-in. (1.9-cm) diameter	400	7	2200
2	Utility Brass Co.	C101	Received as cold-rolled, 1-in. (2.54-cm) diameter	385	8	223
3	Utility Brass Co.	C101	Cold-swaged from received, 1- to 5/8-in. (2.54- to 1.6-cm) diameter	385	8	212
4	Amax Corp.	C101	Cold-swaged from received, 1- to 5/8-in. (2.54- to 1.6-cm) diameter	400	6	233
		C101	Cold-swaged from received, 1- to 5/8-in. (2.54- to 1.6-cm) diameter	385	8	229
5	Stock, free machining	C147	Cold-rolled to 5/8-in. (1.6-cm) diameter			
	(Sample 1)			650	6	200
	(Sample 2)			350	6	110
	(Sample 3)			450	6	135
	(Sample 4)			650	6	156
	(Sample 5)			-	-	75
	(Sample 6)			-	-	75

Note that the RRR's for Groups 2-4 samples were within a relatively narrow range extending from 212 to 233, reflecting the C101 impurity composition and the fully annealed state. For Group 5, however, the RRR values in the annealed condition varied from 110 to 200, reflecting the nonuniform distribution of the second-phase impurities in the free-machining grades.

D. Stress-Strain Cycling Modes

Figure 10 is a schematic stress-strain diagram for a hypothetical specimen subjected to 600 constant-strain cycles of elastic plus plastic strain. The strain remains directly proportional to the stress as long as the stress remains within the "elastic" limit of the specimen. If the stress is removed before plastic yielding, the specimen returns to its original shape and position at zero strain. On an atomic scale, elastic strains do not generate significant quantities of defects and thus do not increase the electrical resistivity of the specimen.

Fig. 10

Stress-Strain Diagram for Group 2 Sample Subjected to 600 Constant-strain Push-Pull Cycles. Strain notations are ϵ_t and ϵ_p , the total strain per cycle and the loop width or plastic strain per cycle at zero stress. Neg. No. MSD-64241.

When cyclic stresses in excess of the elastic limits are applied to a specimen, the stress-strain curve forms a "hysteresis" loop. In this report the loop width at zero stress is defined as ϵ_p , the amount of plastic strain in the cycle. As cycling progresses, the interactions between dislocations and the point-defect production during plastic straining cause work-hardening of the material with a progressive decrease in ϵ_p ; i.e., the stress-strain loop continually narrows during cycling. The specimen quickly approaches a

completely elastic behavior if the cycling continues for several periods at constant load. At constant strain, however, the work-hardening is reflected in the added stress per cycle to produce ϵ_t , and ϵ_p decreases to a constant nonzero value.

The present apparatus makes it relatively easy to simulate any mode of deformation that may be encountered by the copper during operation of a magnet. The cycling modes encountered because of the hoop stresses generated by the internal magnetic fields will depend on the operation and construction of the magnet coils. For economic reasons as well as for weight and space limitations, the construction will probably contain a steel load-bearing hoop, with sufficient steel to ensure that the coil dimensions will return to their original dimensions in the deenergized state. In this situation, the total strain per cycle (ϵ_t) will depend on the Young's modulus and total stress in the steel. The magnitude of ϵ_p in the copper will depend on the cross section of the copper matrix, the magnitude of ϵ_t and the degree of work-hardening in the copper. The latter will most probably be chosen to satisfy the minimum ρ_0 at a reasonable cost, namely, fully annealed C101 OFE copper. The cross section of copper will be minimized to satisfy the allowable ϵ_p , or $\Delta\rho$, that can be permitted for a given ϵ_t .

The primary intent of the present study was to determine the $\Delta\rho$ developed for constant ϵ_t values between 0.10 and 0.30% strains. Before the constant-strain studies were begun with OFE samples, several exploratory runs were carried out with $\Delta\rho$ and work-hardening observations during constant-load cycling of the very pure 5N-grade copper (Table I). The actual mode and amplitudes of stress and strains are presented in Sec. IV below.

The recovery or loss of $\Delta\rho$ by warming was not investigated by a systematic isochronal-annealing study. The results reported here are due to observations made after overnight or several-day warming excursions because of interruption of the cycling experiments. These annealing observations are perhaps sufficient to simulate the recovery that will take place during warming of magnet coils during service. They are clearly insufficient to establish the mobility of the point defects, by comparison with annealing spectra following irradiation damage or single-crystal mechanical deformation.

IV. RESULTS

A. Constant Peak Load

Most of the constant-peak-load experiments were carried out with the Grade 1 copper. Figures 11 and 12 show typical results for RRR's of 3450

Fig. 11. Total Resistivity ρ_0 and ρ_0/ρ_{0i} as a Function of the Number of Constant-load Tension-Compression Cycles at 4.2 K for High-purity Copper with RRR = 3450, Neg. No. MSD-64234.

N curve reflects the rapidly decreasing ϵ_p per cycle as it approaches an apparent saturation value at $N = 1500$. In this case, the $\rho_d = 2.5\rho_{0i}$, where

Fig. 12

Total Resistivity and Fractional Change with (1) Constant-load Cycling to $N = 800$, as in Load-Strain Diagram; (2) Warming to 185 K after $N \approx 800$; (3) Continuation of Cycling at 175-kg Load; and (4) Gradual Increase in Load per Cycle so as to Achieve $\epsilon_t = 0.30\%$. Initial RRR = 700. Neg. No. MSD-64242.

ρ_{0i} is the initial ρ_0 . Seventy-five percent of the total ρ_d is produced by $N = 200$, where $d\rho_0/dN$ decreased rapidly.

Figure 12 shows the effect of warming to 185 K during a 15-h interruption after constant-load cycling of another high-purity sample to ± 175 kg. The vertical dashed line at $N = 800$ indicates recovery during warming of about $1 \text{ n}\Omega\cdot\text{cm}$ in ρ_0 measured at 4.2 K after the annealing. This was about 20% of the total ρ_d . The anneal also evidently caused a strain-aging effect, in which the point defects migrated to the dislocations causing an increase in the yield stress by dislocation pinning. This appears to be the same effect observed by Blewitt et al.¹² and by Birnbaum²⁰ after unidirectional strains and by Hull²¹

and by Johnson and Johnson¹⁷ after cyclic strains. The subsequent increase in ρ_0 in Fig. 12 at $N > 1000$ is the result of increasing the load gradually with N , so as to increase ϵ_t to 0.3%.

Fig. 13

Decrease in Resistivity of Cold-worked C147-grade Copper during Constant-load Cycling at $\epsilon_p = 0$. The elastic-strain "softening" effect decreases with increase in stress. Neg. No. MSD-64236.

$25 \text{ n}\Omega\cdot\text{cm}$; thus the maximum observed decrease in ρ_0 due to elastic cycling is about 10%. The source of this unusual observation is not clear, and it is not known whether it occurs in cleaner material; i.e., this effect may be related to impurity-dislocation interactions, although thermally activated migration is not involved.

B. Load-Unload Cycling

Figure 14 shows ρ_0 versus N with the load-unload cycling of a high-purity copper sample. These data were obtained by introducing a total strain of 0.3% and then cycling between strain limits of +0.3 and +0.1%; the lower strain limit occurred at the zero load. The cycling conditions of the first $1\frac{1}{4}$ cycles are similar to the loop shown in the insert. Most of the increase in ρ_0 occurs in the first quarter-cycle with about 0.18% plastic strain. Subsequent load-unload cycling involved $\epsilon_p < 0.10\%$ and, thus, very little increase in ρ_0 .

A similar rapid saturation was observed in a slightly different cycling mode with load limits of +150 and -50 kg. The width of the loop generated, as

shown in Fig. 15, is smaller than 0.10% strain. Thus, the resistivity of the specimen shows a relatively rapid increase during the first quarter-cycle and a much smaller increase during subsequent cycling as the loop gradually narrows.

Fig. 14

Typical Plot of ρ_0 vs N for Load-Unload Cycling, as Indicated in Load-Strain Diagram. Neg. No. MSD-60548.

Fig. 15

Rapid Saturation in ρ_0 vs N for Cycling Mode with Small Compression Strain after Unloading. Neg. No. MSD-60544.

The simple load-unload cycle for the copper matrix would apply to a magnet structure with no steel-hoop structure and insignificant load bearing by the superconducting filaments. As the amount of steel reinforcement and/or superconductor is increased, the amount of compression in the copper should increase, assuming no plastic yield in the structural or superconductor materials. With sufficient structural material, the compressive strain on unloading will be significant and give rise to wider loops and a greater increase in ρ_0 . In addition, the cold-working in the copper will no longer restrain ϵ_t , since the total strain will approach that in steel subjected to a constant load in the elastic range, i.e., a constant strain.

C. General Characteristics of Constant-peak Strain Cycling Results

The stress-strain plot of Fig. 10 represents the constant-peak strain mode for a C101 sample of Group 2 in which the constant-peak strain, ϵ_t , is 0.20%. Although the stress-strain curve during the initial quarter-cycle is typical of easy-glide behavior, the character of the curve changes rapidly during the first few cycles. The stress to produce $\epsilon_t = 0.20\%$ increases from about 61.25 MN/m² in the fully annealed state to about 102.9 MN/m² during the initial 100 cycles (see Fig. 16). The other rapidly changing parameter is the loop width at zero load, or the ϵ_p per cycle, which decreases from about 0.12% in the first full cycle to 0.055% after $N = 500$.

Fig. 16. Defect Resistivity (left scale), Peak Stress per Cycle (right scale), and Loop Width at Zero Load, ϵ_p , as a Function of Number of Constant-strain Cycles at $\epsilon_t = 0.20\%$ for Group 2 Sample. Neg. No. MSD-64251.

In Fig. 16, the peak stress, the loop width at zero load, and the increase in resistivity ρ_d are plotted as a function of N for this Group 2 sample. Typical data such as Figs. 10 and 16 have the following general properties:

1. The increase in ρ_d has three different stages. For $N < 30$ (Stage I), ρ_d increases rather rapidly with an average value of $d\rho_d/dN = 5.5 \times 10^{-2} \text{ n}\Omega\cdot\text{cm}/\text{cycle}$. The region of $50 < N < 200$ (Stage II) is a transition region from the rapid increase of ρ_d to a linear increase in ρ_d , with an average slope of $d\rho_d/dN = 2.8 \times 10^{-2} \text{ n}\Omega\cdot\text{cm}/\text{cycle}$. For $N > 200$ (Stage III), the increase in ρ_d is linear ($d\rho_d/dN = 1.3 \times 10^{-2} \text{ n}\Omega\cdot\text{cm}/\text{cycle}$ is a constant).

2. Two days' anneal at 300 K after cycling of $N = 500$ is sufficient to anneal out the ρ_d produced in Stage III of the cycling process. After another 100 cycles, one day of annealing at 300 K is not enough to remove the defect production of Stage III. In this sample, the increase in ρ_d after each annealing was linear with the same value of $d\rho_d/dN = 1.3 \times 10^{-2} \text{ n}\Omega\cdot\text{cm}/\text{cycle}$ as before annealing. This behavior indicates that the sample remains in Stage III during the anneal.

3. The peak stress and the loop width at zero load versus N show the work-hardening during the cycling. As with $d\rho_d/dN$, the work-hardening rate is very large in the first 30 cycles and decreases rapidly to a relatively constant rate above $N = 200$. In contrast to $d\rho_d/dN$, there is no clear separation of stages in the work-hardening rate before the first anneal.

4. The anneal-hardening, or strain-aging, effect after 2 days at 300 K is clearly observed by the increase in the peak stress for 0.20% ϵ_t and the decrease in ϵ_p . The progression of work-hardening after the initial anneal

differs from that of ρ_d in that there are virtually no changes in peak stress and ϵ_p after the first annealing and that the subsequent anneals have no significant effect on work-hardening.

D. Effects of Impurities and Fabrication History on Results of Constant-strain Cycling

Figures 17-19 show the results of constant-strain cycling at $\epsilon_t = 0.20\%$ for the different groups of samples listed in Table I. Table II lists the values of $d\rho_d/dN$ at different stages of work-hardening of all samples tested. The increase in ρ_d with N is generally similar to the variation of ρ_d in Fig. 16. For a given N in Fig. 17, ρ_d for different samples can be divided into three different categories: the highest increase is shown in samples from Group 2, the lowest increase in Group 1, and intermediate increases for Groups 3-5.

Although the relatively low rate of increase in ρ_d of the high-purity copper (Group 1) (Fig. 17) suggests that impurity content may be an important factor in explaining $d\rho_d/dN$, the outstanding differences between the Group 2 samples and the Group 3 and 4 samples shows that other factors are of much

Fig. 17

Defect Resistivity, ρ_d , as a Function of Constant-strain Cycles at $\epsilon_t = 0.20\%$ for Eight Samples of the Five Groups Listed in Table I. Neg. No. MSD-64250.

Fig. 18

Stress per Cycle to Produce Constant-strain Amplitude of 0.20% for Samples of Groups 2-5. Neg. No. MSD-64249.

Fig. 19. Progressive Decrease in Loop Width at Zero Load, or ρ_d , with Increase in N for $\epsilon_t = 0.20\%$ in Samples of Groups 2-5. Neg. No. MSD-64244.

TABLE II. Summary of Average ρ_d Increase per Cycle during Constant-strain Cycling, Arranged in Increasing Total Strain per Cycle, ϵ_t

ϵ_t , %	Sample Group	RRR	$\Delta\rho_d/\Delta N$, Units of 10^{-3} n Ω -cm/cycle	
			$0 < N < 30$	$N \geq 30$
0.05	5	75	0	0 (0-8200) ^a
0.10	2	223	0	2.5 (300-550)
	5	156	3	0.5 (300-1600)
0.15	2	223	2.3	2.3 (0-600)
0.175	2	223	6.3	6.3 (0-350)
0.20	1	2200	29	4.1 (150-275)
				6.8 (275-410)
	2	223	55	28 (50-190)
				13 (200-700)
	3	212	39	13 (100-200)
				10 (250-400)
	4	233	34	11 (150-300)
				5.5 (400-700)
	4	236	40	12 (150-300)
				6.5 (400-700)
	4	237	42	13 (150-300)
			36	20 (50-100)
	5	200	45	15 (70-250)
				12 (300-500)
		150	23	16 (80-280)
0.30	2	223	80	26 (200-380)
	3	206	70	20 (150-200)
				14 (300-950)
	4	229	70	12 (500-700)
0.42	5	110	370	100 (70-80)

^aNumbers in parentheses indicate range of ΔN for computing $\Delta\rho_d/\Delta N$.

greater importance among commercial grades. Unlike the sample from Group 2, the five samples from Groups 3 and 4 that were cycled at $\epsilon_t = 0.20\%$ all showed ρ_d values of 6.5 ± 0.5 n Ω -cm at $N = 500$ cycles or about 60% of that in Group 2 samples, even though the starting RRR values were essentially the same. The increase in the peak stress for $\epsilon_t = 0.20\%$, at $N = 500$, is also considerably higher for the Group 2 sample, as is expected for a correlation of ρ_d and work-hardening.

A similar effect of fabrication history is observed for the samples cycled with constant peak strains of 0.30% (see Fig. 20). The first C101 sample cycled at this ϵ_t was from Group 2, whereas the other two samples tested at $\epsilon_t = 0.30\%$ were from Groups 3 and 4, respectively. At $N = 380$, the ρ_d for the Group 2 sample reached $14.5 \text{ n}\Omega\cdot\text{cm}$, whereas for the latter two samples, ρ_d was between 10 and $10.5 \text{ n}\Omega\cdot\text{cm}$. Unlike the case of $\epsilon_t = 0.20\%$, the difference in work-hardening parameters between Group 2 and the other two groups shows up only above $N = 200$ (see Fig. 21).

Fig. 20. Defect Resistivity, ρ_d , as a Function of Constant-strain Cycles at $\epsilon_t = 0.30\%$ for Samples of Groups 2-4. Neg. No. MSD-64248.

The effort to understand the significant differences between Group 2 samples and the other C101 samples involved grain-size studies, spectrochemical analyses of vapors from

oxidized samples, and a cursory search for differences in texture caused by the differences in fabrication before machining. The recrystallized grain sizes in all the C101 rods (Groups 2-4) were all within the range of $0.040 \pm 0.005 \text{ mm}$, with the Group 2 samples at the lower end. The high-purity samples (Group 1), on the other hand, had starting grain sizes varying from 2 to 3 mm. Thus the very large grain size could well account for the relatively low rates of work-hardening and $d\rho_d/dN$ values in the Group 1 high-purity material. However, the small differences in grain size among the C101 materials probably did not influence the final results.

Fig. 21
Stress per Cycle to Produce Constant-strain Amplitude of 0.30% for Samples of Groups 2-4. Neg. No. MSD-64235.

As testimony to the absence of grain-size effects among the commercial samples, the average grain size in the annealed C147 Group 5 samples was significantly larger (0.065 mm) than in the C101 samples. Nevertheless, the ρ_d values at $N > 0$ (see Fig. 17) exceeded those for the Group 3 and 4 samples.

The spectrochemical analyses showed no significant differences in purity among the commercial-grade materials that could account for the observed differences in the results. The largest concentrations of the impurity elements were in the 10- to 15-ppm (by weight) range with total impurity content below 50 ppm.

The texture differences between Group 2 and Group 3 and 4 samples are inferred from a diffractometer scan of reflected X-ray intensities from transverse cross sections of two machined samples in the recrystallized state. The results of this study are shown in Table III, listing the diffracted intensities relative to that of the (111) reflection in a powder sample. For the Group 2 sample, the most significant deviation from the random sample was in the (200) reflected intensity, while the (111) and (222) reflections were about the same intensity as the random case. The Group 3 rod, in contrast, showed a significantly different intensity spectrum, with the (111) and (222) reflected intensities significantly greater than in the random case and with an integrated average deviation from random of 31% compared to 24% for the Group 2 rod.

TABLE III. Reflected Relative Intensities (I) from 2 θ Scan of Plane Perpendicular to Rod Axes of Recrystallized Samples

Indices of Diffraction Planes	Powder I_r (Reference Intensity)	I for Group 2 (Machined from as Received)	I for Group 3 (Swaged to 75% Ra before Machining)
111	100	107	129
200	46	64	43
220	20	10	4
311	17	11	12
222	5	5	8
400	3	3	3
331	9	5	4
420	8	3	5
	208	208	208
$R = \frac{\sum I_r - I }{I_r}$		0.240	0.308

E. Strain-amplitude Effects at Constant Strain per Cycle

The effects of strain amplitude on the change in ρ_d per cycle and on the work-hardening parameters were investigated to some degree over the range of 0.05-0.42%. The primary objective was to determine whether the changes in $d\rho_d/dN$ at higher amplitudes are continuous or catastrophic at $N < 1000$ cycles and to ultimately predict whether any significant effects will occur at high-frequency/low-amplitude conditions.

The experiments at $\epsilon_t = 0.30\%$ were carried out with samples from Groups 2, 3, and 4, respectively, and the data are plotted in Fig. 20. As noted above, these data are similar to the $\epsilon_t = 0.20\%$ data in that the Group 2 sample

is clearly subjected to a faster rate of work-hardening. They are also similar to the data from the 0.20% strain experiments in that $d\rho_d/dN$ gradually decreases with increasing N to nearly constant values at $N > 300$, reflecting the increases in peak stress (see Fig. 21) and the decrease in plastic strain per cycle, noted in Fig. 22. The curves for 0.20 and 0.30% amplitude differ in character from the curve for 0.10% strain amplitude in that, in the latter case, the initially rapid increase in ρ_d and work-hardening parameters are not observed (see Figs. 23-25). At $\epsilon_t = 0.10\%$, the initial plastic strain per cycle is only about 0.02%, so that more than 50 cycles are necessary before a significant change in ρ_0 is observed.

Another unique aspect of the curves for $\epsilon_t < 0.20\%$ (see Fig. 23) is that $d\rho_d/dN$ increases with N for the initial 730 cycles at 0.10% and for the continuation of cycling at 0.150 and 0.175%. The relatively small decrease in ρ_d during the 300 K anneal after both 0.10 and 0.175% strain amplitudes indicates that most of the resistivity is derived from dislocations rather than point defects.

Fig. 22

Decrease in ϵ_p per Cycle for $\epsilon_t = 0.30\%$ in Samples of Groups 2-4. Neg. No. MSD-64240.

Fig. 23
Comparison of Increase in ρ_d with N for Different Constant-strain Amplitudes. Neg. No. MSD-64238.

Fig. 24. Comparison of Peak-stress Increases for Group 2 Samples with Different ϵ_t . Neg. No. MSD-64237.

Fig. 25. Comparison of Plots of ϵ_p vs N for Group 2 Samples with Different ϵ_t . Neg. No. MSD-64239.

The experiment at $\epsilon_t = 0.42\%$ was carried out with a sample from Group 5. The conclusions drawn from this test indicate that defect production during the initial cycling stage at this level of constant strain amplitude is catastrophically larger than at 0.30% . This is shown in Fig. 26 where ρ_d is plotted as a function of strain amplitude. The data for Fig. 26 are all taken from experiments with Group 2 samples, except for the point for the $N = 30$ cycle curve at $\epsilon_t = 0.42\%$. The data indicate that ρ_d is relatively unimportant at $\epsilon_t < 0.15\%$ and that the important contributions begin for $\epsilon_t > 0.175\%$. The data further indicate that the rate of increase in ρ_d with ϵ_t becomes very large above 0.30% . This point should be further investigated, however, especially for the larger N values. Since work-hardening and, therefore, closing of the stress-strain cycling loop should occur rapidly at higher ϵ_t , saturation in ρ_d may be expected to occur at relatively low N values.

Fig. 26

Defect Resistivity as a Function of ϵ_t per Cycle at $N = 30, 200$, and 400 cycles. All data are from Group 2 samples, except for point at $\epsilon_t = 0.42\%$. Neg. No. MSD-64243.

F. High-frequency Cycling at Low Amplitudes

An important objective of this study is to evaluate the possibility of degradation in the magnet conductor caused by high-frequency/low-amplitude stresses that may arise during operation. The frequency of the cross-head cycling movement by the reversal of a screw rotation is considerably smaller than the vibrations that may be encountered by interaction with forces from an ac magnet. However, some of the data for $\epsilon_t < 0.10\%$ appear to be relevant and appropriate for estimating the increase in ρ_d over several orders of magnitude in N .

The data referred to above for $\epsilon_t = 0.10\%$ strain amplitude are relevant with respect to long-term vibrations in that strains of this magnitude may be considered acceptable with respect to fatigue life for copper over 10^6 - 10^8 cycles. The data of Fig. 24 indicate that the stress to maintain constant-strain cycles of 0.10% magnitude will remain in the range of less than 73.5 MN/m^2 over an infinite number of cycles, whereas the minimum fatigue-failure stress decreases to approximately 265 MN/m^2 . The data of Fig. 23, however, indicate that $d\rho_d/dN$ may be sufficiently larger over 10^4 cycles to cause an increase of $25 \text{ n}\Omega\cdot\text{cm}$ or reduce the RRR to that of heavily cold-worked structure.

Further experiments extending N to 10^4 cycles at $\epsilon_t = 0.10\%$ would be advisable to determine whether $d\rho_d/dN$ becomes insignificant at N greater than 1000. Two experiments were carried out with $N > 1000$, but with ϵ_t 's of 0.01 and 0.05% . The first was carried out with the sample of Fig. 16, after

$N = 680$ cycles at $\epsilon_t = 0.20\%$ and a 2-day anneal at 300 K. After 3300 cycles with $\epsilon_t = 0.01\%$, no increase in ρ_d was observed. This is to be expected, since no plastic strain was involved in the already work-hardened sample.

The second test was carried out with a sample from Group 5. The results are summarized in Fig. 27. This annealed sample was first cycled to $N = 50$ cycles at $\epsilon_t = 0.10\%$, with a ρ_d of $0.48 \text{ n}\Omega\cdot\text{cm}$. This is a relatively large ρ_d compared to that observed for the same ϵ_t in the C101 material (Fig. 23) and is an indication that ρ_d may be impurity-sensitive in the 0.10% strain region. After $N = 50$ cycles, the ϵ_t was reduced to 0.05% per cycle for 8200 cycles at a rate of 3 s/cycle. The total ρ_d increase was $0.1 \text{ n}\Omega\cdot\text{cm}$. After $N = 8200$, ϵ_t was increased to 0.10% for five cycles; the ρ_d increase was about the same as in the previous 8200 cycles at 0.05% strain.

Fig. 27

Test of Low-amplitude, High-cycle Fatigue Conditions on One Sample of Group 5. Cycling at $\epsilon_t = 0.10\%$ was interrupted at $N = 50$ cycles by inserting 8200 cycles at $\epsilon_t = 0.05\%$. Effects of interruption were removed by 300 K annealing. Neg. No. MSD-64247.

Although the 8200 cycles at 0.05% strain had no significant effect on ρ_d , it did have a relatively large effect on the peak stress to resume $\epsilon_t = 0.10\%$, as is noted in Fig. 27. It should be emphasized that this is not an anneal-hardening, but was specifically related to the low amplitude cycling.

The 300 K anneal for 5 days after the resumption of $\epsilon_t = 0.10\%$ cycling had no strain-aging effect. The mechanism for this hardening effect apparently differs from the anneal-hardening²¹ caused by 300 K annealing, in that neither warming nor point-defect formation is involved. It appears to be a dislocation-redistribution effect.

G. Constant-strain Cycling of Cold-worked Copper

Two experiments were carried out with samples of Group 4 that were not given a recrystallization anneal after machining and had RRR values of 53. No significant change in ρ_0 was observed after $N = 300$ cycles at $\epsilon_t = 0.20$ or 0.30% . For the 0.20% amplitudes, the deformation was totally elastic with constant peak-stress values of 285 MN/m^2 . For $\epsilon_t = 0.30\%$, the first three cycles involved a small plastic strain.

V. DISCUSSION OF RESULTS

A. Application of Data to Magnet Design

1. Prediction of ρ_d in C101-grade Copper

In this study we have attempted to at least qualitatively survey the increments in ρ_d developed under several possible modes of deformation cycling and to find those variables in the cycling parameters and in metallurgical history that are most effective in determining ρ_d in copper. The basic parameter in determining ρ_d is ϵ_p , the plastic strain per cycle. Figures 23 and 25 indicate that even very small but consistent ϵ_p values of 0.01% ($\epsilon_p = 10^{-4}$) will begin to produce a significant increase in ρ_d after several hundred cycles. If the magnet design is such that constant-strain cycling will indeed result from energizing-deenergizing cycles, the present data can be readily used to predict ρ_d accumulation at ϵ_t levels of 0.10-0.30%, assuming that $d\rho_d/dN$ values remain relatively constant after $N = 300$. This appears to be a reasonably safe assumption for at least the first 3000 cycles at $\epsilon_t = 0.10$ and 0.20%, but this rate may perhaps be decreasing at $\epsilon_t = 0.30\%$ to somewhat lower values than those measured here.

As an aid in estimating ρ_d in C101-grade copper, Table IV lists estimated values of ρ_d for $N = 1000$ and $N = 2000$ cycles for ϵ_t values of 0.10, 0.20, and 0.30%. For C101 material with history similar to Group 2 of Table I, ρ_d can accumulate to ~ 50 n Ω ·cm after 2000 cycles at $\epsilon_t \approx 0.3\%$. For the $\langle 111 \rangle$ texture, however, the same conditions should produce only 29 n Ω ·cm, which is a substantial reduction that can be achieved by careful specification of metallurgical history.

TABLE IV. Defect Resistivities, ρ_d , from Constant-strain Cycling at 4.2 K, as Calculated from Measured $d\rho_d/dN$ Values for C101-grade Copper

	1000 Cycles			2000 Cycles		
$\epsilon_t, \%$	0.10	0.20	0.30	0.10	0.20	0.30
ρ_d (n Ω ·cm) $\langle 100 \rangle$ or random texture	4.1	17.8	23.6	8.56	30.8	51.6
ρ_d (n Ω ·cm) $\langle 111 \rangle$ texture	-	9.0	~ 17.0	-	15.5	29.0

The importance of the ρ_d values listed in Table IV relative to the initial impurity resistivity, ρ_i , is quite large. For example, if ϵ_t is limited to a maximum amplitude of 0.10%, the ρ_d after 2000 cycles will exceed the starting ρ_0 , and the ratio ρ_d/ρ_0 can reach a factor of 7 at $\epsilon_t = 0.30\%$ for 2000 cycles. Relative to transverse magnetoresistance, ρ_m , the indicated ρ_d values are not overwhelming, but are still quite significant. Some examples of the effect of ρ_d on the total ρ_0 , based on

$$\rho_0 = \rho_i + \rho_d + \rho_m$$

are used to illustrate the relative importance during operation of 40- and 100-kG magnetic fields.

For the former case, with $\rho_m \approx 15 \text{ n}\Omega\cdot\text{cm}$, and 1000 cycles at $\epsilon_t = 0.20\%$,

$$\begin{aligned}\rho_0 &= (7 + 17.8 + 15) \text{ n}\Omega\cdot\text{cm} \text{ for } \langle 100 \rangle \text{ texture} \\ &= (7 + 9 + 15) \text{ n}\Omega\cdot\text{cm} \text{ for } \langle 111 \rangle \text{ texture.}\end{aligned}$$

For the $\langle 100 \rangle$ texture, $\rho_d/\rho_0 \approx 0.45$, whereas for the $\langle 111 \rangle$ texture, $\rho_d/\rho_0 \approx 0.31$. At 100 kG, with ρ_m estimated at $\sim 40 \text{ n}\Omega\cdot\text{cm}$, the ρ_d/ρ_0 ratios are still about 0.29 and 0.15. In terms of material (copper), cost, and weight for a given conductor length and maximum allowable electrical resistance heating (I^2R), the volume of copper required, is directly proportional to ρ_0 . A ρ_d/ρ_0 ratio of 0.10 increases the required volume by roughly 10%, if the heat-capacity and surface-area increases are neglected.

2. Application of Work-hardening Data

The data on the increase in tensile stress to produce a given constant strain per cycle are the only quantitative data for copper at 4.2 K and are clearly needed in the evaluation of the stress-strain data for superconducting composites in service. The work-hardening at $\epsilon_t > 0.175\%$ strain/cycle is very significant during the first 200 cycles. One can expect to observe a significant effect on the yield stress and perhaps the overall ϵ_t of the composite for a given constant stress if the volume ratio of steel to copper is relatively small.

The need for these data becomes most apparent in that the increase in work-hardening cannot be predicted from literature data for unidirectional deformation at 4.2 K, as given in Ref. 12 or 13, for example. Although the initial stress values do indeed correspond to the yield-stress values for fully annealed copper, the increases in work-hardening with the number of cycles are considerably smaller than expected from a comparison of stress to total plastic strain, where total ϵ_p is obtained from the summation of ϵ_p over N for all the data. Figure 28 shows the plots of peak stress per cycle versus $\sum \epsilon_p$ for the several cyclic constant-strain experiments carried out in this study. The data plotted represent experiments with samples of Groups 2-4 and ϵ_t values of 0.20 and 0.30%. The data for the Group 2 samples clearly show higher work-hardening stresses than is shown by the Group 3 and 4 samples. Within each group, the data for the two ϵ_t experiments (0.20 and 0.30%) overlap, indicating that the total cyclic strain is indeed the prime factor in determining ρ_d and the stress to produce a given total cyclic strain.

The curve plotted as a dashed line represents unidirectional tensile-stress/tensile-strain data for polycrystalline OFE copper²² tested at 4.2 K. When this curve is compared with the cyclic-strain curves, the difference in work-hardening mechanisms and/or magnitudes becomes clear. Small-amplitude cyclic strains do not produce the hardening that results from continuous unidirectional strain. This is clear in the initial cycling regime, but

the most important difference is in the relatively rapid decrease in the cycling stress-strain slope near $\sum \epsilon_p = 0.10$ and the indications of near saturation at $\sum \epsilon_p$ of 0.30. In contrast, the typical unidirectional stress-strain curves for either polycrystal or single-crystal deformation at cryogenic temperatures show Stage 3 work-hardening beginning at $\epsilon_p > 0.30$. Thus, design data for evaluating strains resulting from magnet stresses should be carefully evaluated with respect to the difference between cyclic and unidirectional deformation.

Fig. 28. Plots of Peak Stress per Cycle, σ_t , as a Function of Accumulated Plastic Strain, $\sum \epsilon_p$, for the Three Different Groups of C101 Samples. Dashed curve is tensile-stress/tensile-strain data,²² Neg. No. MSD-64246.

Another important application of the present data is the relatively unpredictable effect of 300 K annealing on the work-hardening at 4.2 K. Although the increased stress to produce a given strain is in line with previous observations of strain-aging,^{12,17,20,21} the saturation effects after annealing shown in Figs. 18 and 21 are unexpected and not consistent with ρ_d data. The latter data indicate that the rate of point-defect production during deformation after annealing is similar to that before annealing. Further work-hardening is, on the other hand, severely diminished. This is one important effect that shows that ρ_d increase is not simply related to work-hardening.

Another effect of similar importance is the observation that there is an abrupt increase in work-hardening after 77 K annealing, whereas no significant effect on ρ_d is observed. Periodic warming of a magnet to $T > 240$ K apparently will have the effect of not only decreasing ρ_d , but also arresting the rate of work-hardening. Further basic studies of work-hardening in copper and in other metals at 4.2 K are needed to enable reliable prediction of stress-strain response as affected by texture differences and periodic annealing during magnet operation.

B. Application to Basic Studies of Deformation-induced Point Defects

The value of the present results in relation to point-defect research is that they provide a quantitative basis for modeling the point-defect production from the dislocation interactions during cyclic deformation.²³ The two observations of primary importance in this respect are (1) $d\rho_d/dN$ remains constant for a given ϵ_t , after an initial rapid work-hardening, and (2) the fraction of ρ_d that is lost during the 300 K annealing is that part accumulated during the constant $d\rho_d/dN$ stage. A third related factor is that $d\rho_d/dN$ after work-hardening and 300 K annealing appears to be the same as during the pre-anneal cycling.

1. Stages in Defect Production

There thus appear to be essentially two distinct stages of defect production in constant-strain cycling where Stage I is associated with dislocation production and work-hardening by dislocation interactions. This stage corresponds to the dislocation bundling or clustering stage described by Basinski et al.²⁴ At the levels of ϵ_t at 0.20 and 0.30%, the extent of this rapid work-hardening stage is dependent on texture. For the light texture (Group 2), Stage I appears to extend to $N \approx 250$ cycles, whereas the strong $\langle 111 \rangle$ texture provides several substages at $N < 500$ cycles. Stage II is, of course, characterized by the constant rate of point-defect production per cycle. This again is consistent with the metallographic observations of Ref. 24, in which the accumulation of mottled areas between dislocation bundles is described.

The remarkable inference of the present data is that the rate of point defect production per cycle seems to depend only on the strain ϵ_p and is not directly related to the increase in stress to produce ϵ_t . The absence of dependence on stress is similar to what has been reported for some experiments with unidirectional tensile deformation,²³ where $d\rho_d \propto \epsilon^n$. It is clearly not consistent with the more widely accepted Saada relation,²⁵ where

$$\rho_d \approx \int_0^{\epsilon} \sigma d\epsilon.$$

The latter presumes that the strain energy is the important factor and that the point defects are produced by a mobile dislocation cutting through a stationary forest of dislocations.

2. Comparison with Unidirectional Deformation

The difference between the defect production during unidirectional tension and strain-cycling is, however, very significant. This is indicated in Fig. 29, where ρ_d is plotted against cumulative ϵ_p strain during several of the cycling tests with OFE grades. As expected, the cumulative ρ_d for a given

total ϵ_p for Group 2 samples is larger than those for Groups 3 and 4, and the data for $\epsilon_t = 0.20$ and 0.30% strain per cycle give essentially the same cumulative data. Since the stresses to produce $\epsilon_t = 0.30\%$ are considerably larger than those for 0.20% , the near coincidence of the ρ_d -versus- $\sum \epsilon_p$ data is further confirmation that $\rho_d = a\epsilon^n$, as observed in several of the unidirectional experiments.²³

Fig. 29. Total Defect Resistivity, ρ_d , vs Cumulative ϵ_p , $\sum \epsilon_p$, for Constant-strain Cycled C101 Samples (solid lines) and for Single Crystals Pulled in Pure Tension at 4.2 K (dashed lines).¹² Neg. No. MSD-64245.

The data of Blewitt et al.¹² for tensile loading in single crystals of copper gives $n = 2$ and $a = 0.1 \mu\Omega\cdot\text{cm}$, whereas a review of data from polycrystalline samples²³ concludes that $n \approx 3/2$ and $a = 0.05 \mu\Omega\cdot\text{cm}$. The present data for cyclic strains give $\rho_d = 0.024 \mu\Omega\cdot\text{cm} \sum \epsilon_p^{0.82}$ for the Group 2 texture and $\rho_d = 0.017 \mu\Omega\cdot\text{cm} \sum \epsilon_p^{0.71}$ for the $\langle 111 \rangle$ texture. Since the data for Fig. 29 are primarily from the Stage II region, where $d\rho_d/dN$ is nearly constant, the above equations are presumed to be primarily relevant to the point-defect production, ρ_p . Further research is required, however, to establish the extent of defect annealing at 300 K after Stage I deformation, so as to more clearly separate the two contributions to ρ_d . On the basis of the present data, it appears that unidirectional deformation is considerably more effective in point-defect production on a cumulative plastic-strain basis.

3. Defects per Cycle

The basic information that is useful for analysis of the point-defect production process is $d\rho_d/dN$ in the range above the initial work-hardening stage, i.e., $N > 300$ cycles. Since ϵ_p per cycle is relatively constant, the value of $\Delta\rho_d$ for each cycle reflects the mechanism by which defects are

produced. A parameter of principal interest is the number of defects produced during each cycle. For the Group 2 material, we have established that $\Delta\rho_d$ for each cycle at $\epsilon_t = 0.20\%$ is about $0.013 \text{ n}\Omega\cdot\text{cm}$. If we assume that $\Delta\rho_d$ is from point defects only and that 1 at. % Frenkel defects produces $2.0 \text{ }\mu\Omega\cdot\text{cm}$, the concentration of defects per cycle with $\epsilon_t = 0.20\%$ and $\epsilon_p \approx 0.05\%$ is 0.65×10^{-7} . For $\epsilon_t = 0.30\%$ and $\epsilon_p \approx 0.10\%$, $\Delta\rho_d$ per cycle is $2.6 \times 10^{-5} \text{ }\mu\Omega\cdot\text{cm}$, or about 1.3×10^{-7} defect concentration. For the $\langle 111 \rangle$ textured samples, the defect concentrations per cycle are about half those of the Group 2 samples. These concentrations are consistent with the conclusions²³ from unidirectional-deformation data, where it has been concluded that $\epsilon_p = 1\%$ produces a point-defect concentration of 10^{-6} and $\epsilon_p = 10\%$ gives 10^{-5} .

4. Future Research

The objective of future basic research in this program is to proceed from the present quantitative data to a physical model for the production of point defects by dislocation interactions.

To correlate the ρ_d information with specific kinds of either stationary-obstacle interactions or moving-jog mechanisms, cyclic push-pull strain measurements on single crystals would be of greater value than the unidirectional data, which contain a summation of ρ_d from a continuous increase in both dislocations and point defects. The present data indicate that constant $d\rho_d/dN$ values should be achieved relatively quickly in multiple-glide single crystals and that the $\Delta\rho_d$ per cycle will be very much dependent on the axial orientation.

Another principal objective of future research will be to understand the anneal-hardening effect that is so apparent. Isochronal-annealing studies should consider not only the loss in ρ_d , but also careful measurements of critical resolved shear stresses for constant-strain cycling.

A third area of interest is the extension of these systemic constant-strain cycle measurements to aluminum, which is of considerable interest as a cryogenic electric-current conductor. Experiments on aluminum²⁶ that has been deformed unidirectionally at 20 K show rather sharp annealing spectra, in contrast to that for copper. Thus, the constant-strain cycling at 4.2 K should produce simpler and perhaps even more reproducible ρ_d -versus- N data than in copper.

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REFERENCES

1. Amax Corporation, New York (1977).
2. D. B. Montgomery, J. E. Williams, N. T. Pierce, R. Weggel, and M. J. Leupold, *Adv. Cryogenic Eng.* 14, 88 (1969).
3. J. G. Hust and P. J. Giaratano, *Semi-annual Report on Materials Research in Support of Superconducting Machinery*, NBSIR74-393, p. 32 (Oct 1974).
4. M. Kohler, *Ann. Physik* 32, 211 (1938).
5. H. Brechna, *Superconducting Magnetic Systems*, Springer-Verlag, New York (1973).
6. J. G. Rider and C. T. B. Foxon, *Phil. Mag.* 16, 1133 (1966).
7. T. H. Blewitt, R. R. Coltman, R. E. Jamison, and J. K. Redman, *J. Nucl. Mater.* 2, 277 (1960).
8. H. G. Cooper, J. S. Koehler, and J. W. Marx, *Phys. Rev.* 94, 496 (1954).
9. L. M. Clarebrough, R. L. Segall, M. H. Loretto, and M. E. Hargreaves, *Phil. Mag.* 9, 1377 (1964).
10. J. Molenaar and W. H. Arts, *Nature* 166, 690 (1950).
11. M. J. Druyresteyn and J. A. Manintveld, *Nature* 168, 868 (1951).
12. T. H. Blewitt, R. R. Coltman, and J. K. Redman, in *Defects in Crystalline Solids*, The Phys. Soc. London (1955).
13. W. B. Pearson, *Phys. Rev.* 97, 666 (1955).
14. T. H. Blewitt, R. R. Coltman, C. E. Klabunde, and T. S. Noggle, *J. Appl. Phys.* 28, 639 (1957).
15. J. W. Corbett, R. B. Smith, and R. M. Walker, *Phys. Rev.* 114, 1452 (1960).
16. J. A. Manintveld, *Nature* 169, 623 (1952).
17. E. W. Johnson and H. H. Johnson, *Trans. AIME* 237, 1333 (1965).

18. J. Polak, Czech. J. Phys. *B19*, 315 (1969).
19. Application Data Sheet, Copper Development Association, New York.
20. H. K. Birnbaum, J. Appl. Phys. *34*, 2175 (1963).
21. D. Hull, Phil. Mag. *3*, 513 (1958).
22. K. A. Warren and R. P. Reed, NBS Monograph *63*, U. S. Government Printing Office, Washington, D.C. (1963).
23. A. van den Beukel, *Vacancies and Interstitials in Metals*, W. Schilling et al., eds., North Holland Publishing Company, p. 427 (1969).
24. S. J. Basinski, Z. S. Basinski, and A. Howie, Phil. Mag. *19*, 899 (1969).
25. G. Saada, Acta Met. *9*, 166 (1961).
26. C. Frois, Acta Met. *14*, 1325 (1966).