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**ATHERMIC RELAXATION PHENOMENON  
IN POLYCRYSTALLINE COPPER  
AFTER COOLING  
TO HELIUM TEMPERATURES**

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## INTRODUCTION

Unusual relaxation phenomena of heat release have been recently observed in polycrystalline copper<sup>/1-3/</sup>, amorphous LaZn alloy<sup>/4,5/</sup> and plexiglass<sup>/6/</sup> upon cooling to helium and subhelium temperatures. These phenomena are characterized by extremely long time constants. While the materials mentioned above differ in structure, it seems probable that the relaxation phenomena reported by various research groups<sup>/1-6/</sup> have a common origin. This assumption can be supported by the following facts:

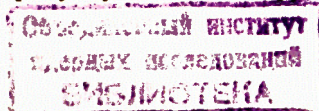
i) in all the cases a simple exponential law  $W(t) = W_0 \exp(-t/\tau)$  describes release of power  $W$  as a function of time  $t$ , where the time constant  $\tau$  ranges from 60 to 150 hours for different copper samples,  $\tau$  equals 55 hours for LaZn and 340 hours for plexiglass; it is noteworthy that the  $\tau$ 's measured for various materials are of the same order of magnitude;

ii) from the "usual" point of view  $W(0)$  are also equally extremely small for all the cases,  $0.5 \text{ nW}\cdot\text{g}^{-1}$  for Cu,  $16 \text{ nW}\cdot\text{g}^{-1}$  for LaZn, and  $0.6 \text{ nW}\cdot\text{g}^{-1}$  for plexiglass; however, the  $W(0)$  values may differ for various samples of the same material, e.g., these differences may sweep three orders of magnitude for copper samples;

iii) the relaxation rate is independent of temperature  $T$  below 50K at least for Cu<sup>/2/</sup> and below 4.2K for LaZn<sup>/4/</sup>. This effect is evidently of great interest both for theory dwelling on its athermic or quantum nature and for practice of reaching ultra-low temperatures upon demagnetization and heat capacity measurements as well as for design of highly sensitive heat detectors.

Two hypotheses have been proposed on the nature of these relaxation phenomena: the orthopara conversion of molecular hydrogen trapped in the sample for LaZn<sup>/5/</sup> and the inelastic relaxation of thermoelastic stresses on grain boundaries for copper<sup>/1,2/</sup>. In spite of the attractive grace of the orthopara conversion idea, there are two serious objections. Firstly, hydrogen diluted in metals exists only in atomic, not molecular form<sup>/7/</sup>, and secondly, the atomic concentration needed for observed  $W(t)$  explanation amounts to 0.1% as follows from the author's calculation and is too great for the case with no special treatment.

In the present paper an attempt is made to estimate the observed value of  $W(0)$  for polycrystalline copper based on the



Table

Values of parameters for expression

sample	1	2	3	$t_1$ , annealed
$W_1, \text{pW/g}$	500	500	0.4	5
$W_2, \text{pW/g}$	50	-	-	-
$\tau_1, \text{h}$	70	75	100	70
$\tau_2, \text{h}$	700	>700	-	-

idea of inelastic relaxation of thermoelastic stresses. For the occurrence of thermoelastic stresses upon cooling the sample should be nonhomogeneous in its thermal properties, e.g., thermal expansion coefficients tensor  $\alpha_{ij}(\bar{r}) \neq \text{const}$ , where  $i, j = 1, 2, 3$ . Really it is so in any sample even in a single crystal, where  $\alpha_{ij}$  (surface)  $\neq \alpha_{ij}$  (volume) and, besides, dislocations can also produce the local fluctuations of  $\alpha_{ij}$ . The polycrystalline copper was chosen for  $W(0)$  estimation because of its simple model structure and the most detailed observation of the phenomenon under discussion was made independently by several groups. As for us we have investigated three samples from different copper but approximately the same purity 99.98. Sample No.1 was made of cast rapidly crystallized copper, No.2 of rolled copper and No.3 of cast copper with unknown conditions of crystallization. For all of them the relaxing power of dissipation is well approximated by the expression  $W(t) = W_1 \exp(-t/\tau_1) + W_2 \exp(-t/\tau_2)$ . The parameters of this expression for  $T = 1\text{K}$  are listed in the Table. It has been shown that the phenomenon is fully reproducible after reheating to room temperature and the two year storing at this temperature does not produce much effect. The results obtained for sample 1 are illustrated in fig.1. In 1980 the measurement was carried out at different temperatures from 4.2K to 1K and proved the athermic character of relaxation<sup>12/</sup>. The fact that there are at least two time constants, not one, is of particular interest.

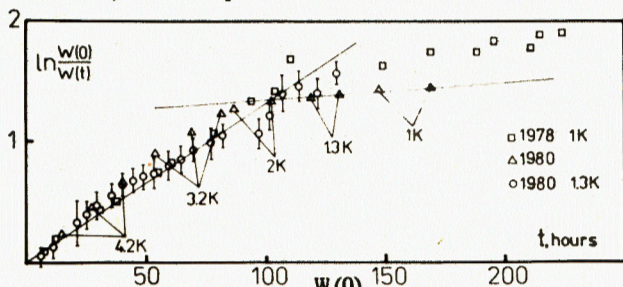


Fig.1. Time dependence of  $\ln \frac{W(0)}{W(t)}$  measured for sample 1 in adiabatic calorimeter.  $W(0)$  is the initial power dissipated per unit mass after the first thermal switch interruption.  $\square$  - measurement of 1978 at  $T = 1\text{K}$ ;  $\Delta$  - measurement of 1980 at  $T = 4.2 \div 1\text{K}$ ;  $\circ$  - 1980,  $T = 1.3\text{K}$  after 6 hr annealing at  $900^\circ\text{C}$  in vacuum of  $10^{-3}$  mm mercury pressure. Solid lines correspond to  $\tau$  equal 75 and 700 hours.

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## CALCULATION

For calculation of  $W(0)$  let us limit ourselves to the case of isotropic grains and boundaries with scalar thermal expansion coefficients  $\alpha_g(T)$  and  $\alpha_b(T)$ , respectively, and two elastic parameters: Young's modulus  $E$  and Poisson's ratio  $\nu$  independent of  $T$  and the same for grains and boundaries. In addition, we have  $b \gg a \gg d$  where  $b, a$  are the characteristic dimensions of the sample and a grain, respectively,  $d$  is the boundary thickness. For our samples with masses from 10 to 25 kg this is without doubt true. Thus we have two small parameters  $\frac{a}{b}$ ;  $\frac{d}{a} \ll 1$ . The estimation is performed within two steps. Firstly, the thermoelastic stresses arising after cooling from  $T_c$  to  $T_1$  are calculated on the basis of the temperature stresses compensation (TSC) method<sup>8/</sup> and then  $W(t)$ , as follows from the formal model of inelasticity<sup>9/</sup>, is found. To estimate the thermoelastic stresses by TSC-method, one need to separate mentally the sample into boundaries and grains and to apply to their surfaces the normal forces

$$\frac{E_u}{1-2\nu} \int_{T_0}^{T_1} \alpha_b dT \quad \text{and} \quad \frac{E_u}{1-2\nu} \int_{T_0}^{T_1} \alpha_g dT, \quad (1)$$

respectively, which remove the temperature strain and produce the hydrostatic pressure  $\sigma_{ij} = \sigma \cdot \delta_{ij}$  in grains and boundaries

$$\frac{E_u}{1-2\nu} \cdot \int_{T_0}^{T_1} \alpha_g dT \quad \text{and} \quad - \frac{E_u}{1-2\nu} \cdot \int_{T_0}^{T_1} \alpha_b dT, \quad (2)$$

respectively, where  $\delta_{ij}$  is the Kronecker beta-symbol;  $i, j = 1, 2, 3$ ;  $E_u$  is unrelaxed Young's modulus. After this the grains are "glued" with the boundaries and the surface normal pressure

$$\frac{E_u}{1-2\nu} \cdot \int_{T_0}^{T_1} \alpha_g dT \quad \text{or} \quad \frac{E_u}{1-2\nu} \int_{T_0}^{T_1} \alpha_b dT \quad (3)$$

is applied to the regions of sample surface occupied by grains or boundaries respectively and the same volume forces

$$\frac{E_u}{1-2\nu} \int_{T_0}^{T_1} \alpha_g dT \quad \text{and} \quad \frac{E_u}{1-2\nu} \int_{T_0}^{T_1} \alpha_b dT \quad (4)$$

which are distributed normally along the surfaces of grain-boundary separation as in fig.2. Because of their opposite direction

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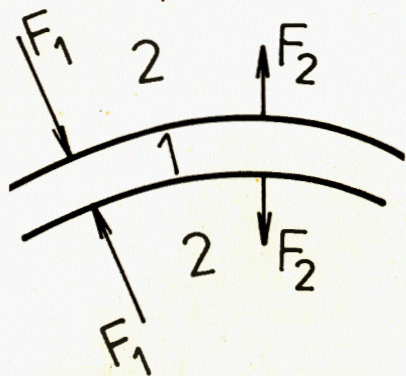


Fig.2. On boundary stress calculation. 1 - boundary, 2 - grains. The resulting normal force density in each point of interface is equal to  $F_1 - F_2$ . The sign "plus" corresponds to stretching while "minus" corresponds to compression.

the resulting force is

$$\frac{E_u}{1-2\nu} \int_{T_0}^{T_1} (\alpha_b - \alpha_g) dT. \quad (5)$$

The stresses produced by the surface (3) and volume (5) forces together with (2) lead to the true thermoelastic stresses. As a result we have now a typical problem of the theory of elasticity. Further for the sake of simplicity, it is supposed that the inelasticity is connected with the boundaries only because of their greater disorder as compared with the grains. That is why it is enough to find the boundary stresses. Let us consider the central part of the sample. Each grain can be approximated by the polyhedron. If we introduce the local Cartesian basis  $\{XYZ\}$  on the boundary with  $Z \parallel \bar{n}$ , where  $\bar{n}$  is the unit vector normal to the boundary, and the axes X and Y are in the boundary plane then it follows from Saint-Venant's principle that only two components of  $\sigma_{ij}$  exist

$$\sigma_n \equiv \sigma_{ZZ} \approx \text{const} \quad \text{and} \quad \sigma_t \equiv \sigma_{XX} = \sigma_{YY} \approx \text{const}, \quad \sigma_{XY} \equiv \sigma_{nt} = 0. \quad (6)$$

This is not correct in the regions with the dimensions of the order of  $d$  near the polyhedron edges but their contribution to the relaxation is small. Furthermore we have  $\sigma_t \approx 3\sigma_n$  and

$$\sigma_t = -\frac{E_u}{1-2\nu} \int_{T_0}^{T_1} \alpha_g dT \quad (\text{appendix A})$$

so only the tangential component  $\sigma_t$  relaxation is taken into account below. Then the power dissipated in the unit volume of the boundary (appendix B) is

$$W_b(t) \approx \frac{1}{(1-2\nu)^2} \cdot \left[ \int_{T_0}^{T_1} \alpha_g dT \right]^2 \cdot \frac{2+\Delta}{1+\Delta} \cdot \frac{\delta E}{r} \cdot \exp(-t/\tau), \quad (7)$$

where  $\delta E = E_u - E_R$  is Young's modulus relaxation;  $E_R$  is relaxed Young's modulus;  $\Delta \equiv \delta E/E_R$  is the relaxation degree. The volume of the boundaries can be expressed as

$$V_b \approx 3V_0(d/a), \quad (8)$$

where  $V_0$  is the sample volume. Using the sample mass  $M = \rho V_0$  (where  $\rho$  is density) and relations (7) and (8) we get at last the power dissipated in the unit mass of the sample

$$W(t) \approx \frac{3}{(1-2\nu)^2} \left[ \int_{T_0}^{T_1} \alpha_g dT \right]^2 \cdot \frac{2+\Delta}{1+\Delta} \cdot \frac{\delta E}{\rho r} \cdot \frac{d}{a} \cdot \exp(-t/\tau). \quad (9)$$

For quantitative estimation let us substitute the values  $\nu = 0.3$ ;  $\rho = 8 \cdot 10^6 \text{ g} \cdot \text{m}^{-3}$ ;  $E = 1.1 \cdot 10^{11} \text{ N} \cdot \text{m}^{-2}$  to expression (9). A well-known  $\alpha(T)$  for polycrystalline copper can be used to evaluate

$$\text{the integral } \int_{T_0}^{T_1} \alpha_g dT. \quad \text{As a result, we get } \int_{300\text{K}}^{1\text{K}} \alpha_g dT = -3 \cdot 10^{-3}.$$

The time constant  $\tau$  is of the order of  $10^2$  hours in our case. From macroscopic grain-boundary relaxation experiments it is known that  $\Delta_{Cu} \approx 0.15^{9/}$ . For our microscopic thermoelastic stress relaxation in boundaries we can conclude that  $\Delta_{Cu} < \Delta < 1$ . We choose  $\Delta = 0.2$ . Using the value  $10^{-3} - 10^{-4}$  for  $\frac{d}{a}$  ratio which is unknown for our experiment and substituting to (9) all the quantities mentioned above we obtain  $W(0) = (2 \div 0.2) \text{ nW} \cdot \text{g}^{-1}$  which agrees well with the observed values of  $W(0)$ . Finally it is necessary to multiply this value of  $W(0)$  by coefficient  $\gamma < 1$ . This follows from the fact that at the initial step of cooling the relaxation is thermally activated and, as a result, the time constant  $\tau$  is small and the unrelaxed thermoelastic stresses  $\sigma_u$  are not frozen till the temperature reaches  $T_f$  as can be seen in fig.3. This remark is not of great importance, of course, if we are interested in the order of  $W(0)$  only, but is essential for the physics of this phenomenon. According to formula (9) the value of  $W(0)$  for the chosen material is determined largely by the unknown quantities  $\Delta$  and  $\frac{d}{a}$ . However, the parameter  $\Delta$  is of minor importance, so if the orders of magnitude of  $\frac{d}{a}$  are roughly estimated the effect of variation in  $\Delta$ , as well as in  $\frac{a_g}{a_b}$  ratio, can be ignored. The value of  $\frac{d}{a}$  ratio can be easily changed by thermal treatment of the sample. We have decreased  $W(0)$  by two orders of magnitude after annealing sample 1 at  $900^\circ\text{C}$  for about 6 hr in vacuum of  $10^{-3}$  mm mercury pressure. This fact supports the initial idea. It is interesting to note that the time constants were not affected by annealing as illustrated in fig.1.

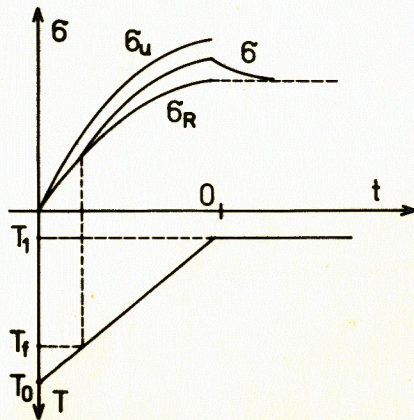


Fig. 3. Probable time dependence of thermoelastic stress  $\sigma$  upon cooling.  $\sigma_u$  and  $\sigma_R$  correspond to unrelaxed and relaxed stresses respectively.  $T_f$  is the "freezing" temperature of non-equilibrium thermoelastic stress. Zero time corresponds to the beginning of relaxation phenomenon observation.

#### CONCLUSION

We have shown that the suggested inelastic relaxation of thermoelastic stresses can provide an energy balance of the phenomenon observed in polycrystalline copper after cooling. A certain assumption on the statistical feature of the  $a_{ij}(\bar{r})$ -field should be made for the same estimation of  $W(0)$  in amorphous solids. From microscopic point of view the relaxation is, possible, due to the tunneling between the two adjacent energy levels similar to the case of the time-dependent specific heat of glasses<sup>11</sup>. However, there are only few discrete time constants in our case. If the tunneling particles are the point defects which form the elastic dipoles, then the heat relaxation phenomenon can be explained by the existence of several non-equivalent orientations in the thermoelastic stress field.

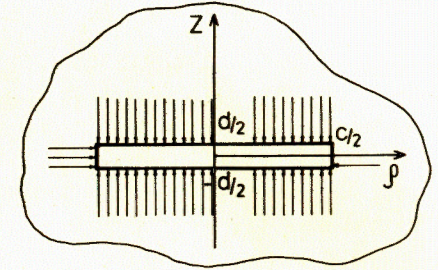
#### APPENDIX A

##### Calculation of $\sigma_n$ and $\sigma_t$

Let us consider a part of the grain boundary at the centre of the sample in the form of a circular plate with thickness  $d$  and diameter  $C$  with  $d \ll c \ll a$  (fig. 1A). For thermoelastic stresses to be evaluated one need to calculate the stresses produced by the forces normal to plate surface which equal

$$\frac{E_u}{1-2\nu} \int_{T_0}^{T_1} (a_b - a_g) dT. \quad (1A)$$

Fig. 1A. A part of boundary in the centre of the sample can be considered as a thin disk in an infinite elastic space with the resulting normal force density  $\frac{E_u}{1-2\nu} \int_{T_0}^{T_1} (a_b - a_g) dT$  applied to the interface. The axes correspond to cylindrical coordinates.



The sum of this stress with hydrostatic one for boundaries (2), stress produced by forces (3) and the contribution of the rest of the sample volume due to forces (4) is the true thermoelastic stress. According to (6) it is enough to evaluate the stress due to (1A) in the centre of the plate  $z=\rho=0$  (in cylindrical coordinates). In Cartesian coordinates  $\{x_i\}$ , where  $i = 1, 2, 3$ , Green's tensor for unit force applied at  $(0, 0, 0)$  along the  $X_k$  axis is<sup>10</sup>:

$$\sigma_{kij} = \frac{\lambda + \mu}{4\pi(\lambda + 2\mu)} \left\{ 3 \frac{x_k x_i x_j}{r^5} + \frac{\mu}{\lambda + \mu} \cdot \frac{\delta_{ki} x_j + \delta_{kj} x_i - \delta_{ij} x_k}{r^3} \right\}, \quad (2A)$$

where  $\lambda, \mu$  are the Lamé coefficients;  $r$  is the distance between the point  $(0, 0, 0)$  and the point of observation. Then using (2A) and (1A) one finds that the surface element  $\rho d\rho d\phi$  produces at the centre the stress

$$d\sigma'_{zz} = \frac{\lambda + \mu}{\pi(\lambda + 2\mu)} \left\{ \frac{3d^3}{(4\rho^2 + d^2)^{5/2}} + \frac{\mu}{\lambda + \mu} \cdot \frac{d}{(4\rho^2 + d^2)^{3/2}} \right\} \times \frac{E_u}{1-2\nu} \cdot \left[ \int_{T_0}^{T_1} (a_b - a_g) dT \right] \rho d\rho d\phi. \quad (3A)$$

After integration of (3A) over the surfaces  $z = \pm \frac{d}{2}$  and substitution of  $\lambda = \nu E / (1 + \nu)(1 - 2\nu)$  and  $\mu = E / 2(1 + \nu)$  one obtains, to a zero-order in  $\frac{d}{c}$  approximation, that the forces (1A) produce the normal stress

$$\sigma'_n = \sigma'_{zz} = - \frac{E_u}{1-2\nu} \cdot \frac{5-4\nu}{8(1-\nu)} \cdot \int_{T_0}^{T_1} (a_b - a_g) dT. \quad (4A)$$

The factor  $(5 - 4\nu)/8(1 - \nu)$  is slightly affected by  $\nu$  when  $0.2 < \nu < 0.4$  and is taken to be  $2/3$  below. Now a reasonable assumption should be made about correlation between  $a_b(T)$  and  $a_g(T)$ , which is, unfortunately, unknown for copper. It is only

evident that  $a_b(T) > a_g(T)$ . On the other hand for single-crystal silicon, for example, it is known that  $a$  (surface) is 2-8 times greater than  $a$  (volume)<sup>12/</sup>. It is supposed that the sample surface and grain boundary are, probably, similar two-dimensional defects. Let us take  $a_b(T) = 2a_g(T)$ . The justification of such assumption is that the effect of variation of  $a_b/a_g$  ratio is not of great importance as compared with the uncertainty of the quantity  $\frac{d}{a}$ . Now we have

$$\sigma'_n \approx -\frac{2}{3} \cdot \frac{E_u}{1-2\nu} \cdot \int_{T_0}^{T_1} a_g dT. \quad (5A)$$

As follows from the Saint-Venant principle the forces (3) induce in the sample a hydrostatic stress

$$\sigma''_{zz} = \sigma''_{yy} = \sigma''_{xx} = \frac{E_u}{1-2\nu} \int_{T_0}^{T_1} a_g dT. \quad (6A)$$

It is easy to demonstrate that the background stress contribution from the rest of the sample volume due to the forces (4) is of the order of  $(\frac{a}{b})(\frac{d}{a})^2 \ll 1$  and can be ignored in  $\sigma_n$  and  $\sigma_t$  estimation. Finally, summing (6A) with (5A) and (2) we have the normal component of the boundary stress to be

$$\sigma_n = -\frac{1}{3} \cdot \frac{E_u}{1-2\nu} \cdot \int_{T_0}^{T_1} a_g dT. \quad (7A)$$

The same procedure with the use of (2A) can be performed for calculating the tangential component  $\sigma_t$  produced by the forces (1A) applied to the surface  $\rho = \frac{c}{2}$ . As a result we get that  $\sigma'_t \sim \frac{d}{c}$  and the thermoelastic  $\sigma_t$  is determined largely by (6A) and (2), that is

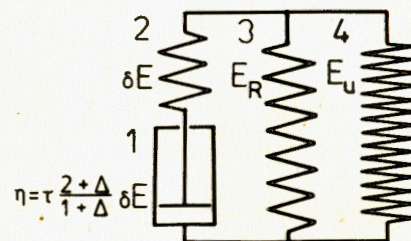
$$\sigma_t = -\frac{E_u}{1-2\nu} \int_{T_0}^{T_1} a_g dT. \quad (8A)$$

## APPENDIX B

### Dissipated Power Evaluation

It is well known that the relaxation of stress  $\sigma$  (strain  $\epsilon$ ) when the constant  $\epsilon_0$  ( $\sigma_0$ ) is applied is described by the simple

Fig.1B. Modified Maxwell's model of inelastic solid for thermoelastic stress relaxation representation.



models of Maxwell or Voigt<sup>9/</sup>. In our case we cannot assume  $\sigma$  or  $\epsilon$  to be constants, since they relax simultaneously. To take this into account, one needs to add to the simple models mentioned above one more spring as fig.1B shows, here  $\eta$  denotes the Newtonian viscosity of the damper 1,  $E_r$ , which is equal to relaxed Young's modulus, denotes the stiffness coefficient of the spring 2 and so on. In the initial state the springs 2 and 3 are stretched out while the spring 4 is compressed. This is the case of a rapid temperature change, when the thermoelastic stress  $\sigma_0 = -\frac{E}{1-2\nu} \int_{T_0}^{T_1} a dT$  occurs. The following relations

for the elements 1,2,3 can be written:

$$\begin{aligned} \sigma_1 &= \sigma_2 = \eta \dot{\epsilon}_1, \\ \sigma_2 &= \delta E \cdot \epsilon_2, \\ \sigma_3 &= E_R \cdot \epsilon_3, \\ \sigma_2 + \sigma_3 &= \sigma, \\ \epsilon &= \epsilon_3 = \epsilon_1 + \epsilon_2. \end{aligned} \quad (1B)$$

And for the fourth element the equation is

$$\sigma = -\sigma_4 = \sigma_0 - E_u \epsilon. \quad (2B)$$

After replacing  $\epsilon_1$  and  $\epsilon_2$  in the last relation (1B) by their expressions through  $\epsilon$  and  $\sigma$  one obtains the initial state relaxation equation

$$E_R \epsilon + r' E_u \dot{\epsilon} = \sigma + r' \dot{\sigma}, \quad (3B)$$

where  $r' = r(2+\Delta)/(1+\Delta)$ ;  $\Delta = \delta E/E_R$ .

Substituting to (3B)  $\sigma$  from (2B) one can rewrite (3B) in the form

$$\epsilon + 2r' \dot{\epsilon} = \sigma_0 / (E_R + E_u). \quad (4B)$$

Complete solution of the equation (4B) can be written in the form

$$\epsilon(t) = A_0 \sigma_0 / (E_R + E_u) + A_1 \exp(-t/2\tau),$$

where the constants  $A_0$  and  $A_1$  can be determined from the boundary conditions  $\epsilon(0) = 0$  and  $\sigma(\infty) = -\frac{E_R}{1-2\nu} \int_{T_0}^{T_1} \alpha dT$ . Then we have

$$\epsilon(t) = \frac{\delta E}{E_u^2} \cdot \sigma_0 \cdot [1 - \exp(-t/2\tau)]. \quad (5B)$$

The dissipated power per unit volume is  $W = \eta \dot{\epsilon}_1^2$ . It follows from

(1B) that  $\dot{\epsilon}_1 = \dot{\epsilon} \frac{2E_u}{\delta E}$ . Finally after easy transformations one

obtains:

$$W(t) = \frac{\delta E}{E_u^2} \cdot \sigma_0^2 \cdot \frac{2 + \Delta}{1 + \Delta} \cdot \frac{1}{\tau} \cdot \exp(-t/\tau). \quad (6B)$$

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8.	Cryogenics
9.	Accelerators
10.	Automatization of data processing
11.	Computing mathematics and technique
12.	Chemistry
13.	Experimental techniques and methods
14.	Solid state physics. Liquids
15.	Experimental physics of nuclear reactions at low energies
16.	Health physics. Shieldings
17.	Theory of condensed matter
18.	Applied researches
19.	Biophysics

Трофимов В.Н.

E8-83-300

Атермическая релаксация в поликристаллической меди после охлаждения до гелиевых температур

Выдвигается предположение о том, что релаксационное выделение тепла с большими постоянными времени, наблюдаемое в различных типах твердых тел после охлаждения, вызвано неупругой релаксацией термоупругих напряжений. Основанная на этом оценка выделяющейся мощности в поликристаллической меди согласуется с экспериментом.

Работа выполнена в Лаборатории ядерных проблем ОИЯИ.

Препринт Объединенного института ядерных исследований. Дубна 1983

Trofimov V.N.

E8-83-300

Athermic Relaxation Phenomenon in Polycrystalline Copper after Cooling to Helium Temperatures

The long-time heat release relaxation phenomenon observed in different types of solids after cooling is attributed to the inelastic relaxation of thermoelastic stresses. Under this assumption an estimate of the relaxing power for polycrystalline copper is performed which agrees with the observed values.

The investigation has been performed at the Laboratory of Nuclear Problems, JINR.

Preprint of the Joint Institute for Nuclear Research. Dubna 1983