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# A NEW METHOD OF DEFORMATION CALORIMETRY APPLIED TO COPPER SINGLE CRYSTALS*

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## ABSTRACT

A new method of deformation calorimetry is described by which the change of stored energy with deformation is measured. For the determination of the dynamic energy balance of plastic deformation, we deform metal single crystals by tension and measure the applied load $K(t)$, the effective deformation rate $\dot{E}_{exp}$ and the temperature $T(t)$ of our specimen with high accuracy during deformation. The load is determined by using a reference signal (load-fixpoint), the deformation rate by means of a tensiometer which records the strain directly at the crystal. Measurement of specimen temperature is carried out with the aid of micro-thermistors of high temperature resolution, $\Delta T \cong 2 \cdot 10^{-5}$ K, and time resolution $\Delta t \leqslant 0{,}1$ s. An essential feature of our method is the procedure developed for evaluation of the $T(t)$-curves (which are severely influenced by unavoidable heat conduction) using the thermoelastic effect. It works by means of the response function of heat conduction which we record during elastic deformation of the specimen.

Some results are given on the stored energy and its change of a copper single crystal deformed at room temperature.

## 1. INTRODUCTION

### *Plastic deformation*

A well-known experimental procedure for the investigation of the deformation behaviour of solids is the tensile test, where a cylindrical specimen is elongated under the influence of an external load. During the test the load $K$ and the elongation $L - L_0$ ($L_0$ = initial specimen length) are measured (Fig. 1a). These measured quantities are evaluated for stress $\sigma$ = load/cross-section of specimen and relative strain $\varepsilon = (L - L_0)/L_0$ and represented in stress–strain curves (Fig. 1b).

There are two different kinds of deformation processes.

(1) Elastic deformation: the specimen elongates proportional to the increasing

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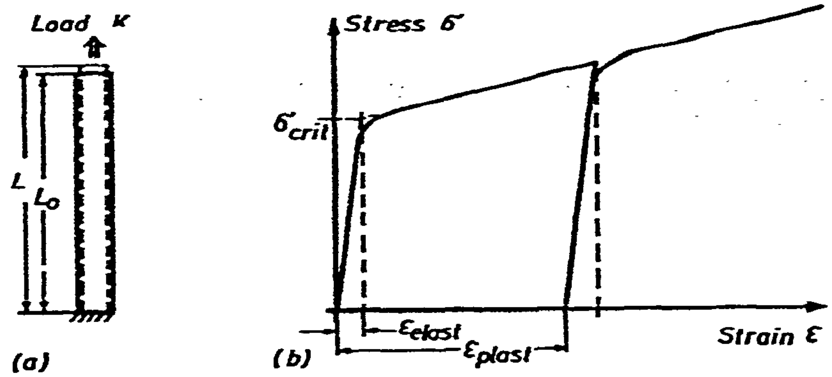


Fig. 1. Tensile test (a), stress-strain curve (b).

stress (Hooke's law) and retains its initial length when the stress is removed (reversible deformation).

(2) Plastic deformation: above a certain critical stress $\sigma_{crit}$ the specimen begins to strain irreversibly. During unloading the plastic part of deformation, which can exceed that of elastic deformation by some orders of magnitude, remains unchanged. A simple analytic stress–strain relation is not available.

The fundamental microscopic process for the deformation of metals and other crystalline solids is the generation and movement of line-shaped imperfections of the crystal lattice, called dislocations. The increase of lattice-defect concentration which is caused by plastic straining leads to a growing resistance against additional deformation. This effect is called work-hardening.

## *Calorimetry*

The changes in concentration and arrangement of dislocations during plastic deformation are associated with a change in energy content of the specimen. Since the energy balance of physical processes is a means to understand them, we use a calorimetric method to measure the change of enthalpy $\Delta H$ during deformation. In metals, about 90% of the work of deformation are converted into heat, 10% remain in the crystal as stored energy.

Two methods are employed for measuring energy storage of plastic deformation: annealing and deformation calorimetry[1]. Here, we report on the application of a new method of deformation calorimetry[2, 3]. The energy stored in plastically deformed crystals, $E_{stor}$, is determined by means of deformation calorimetry as the difference of the work of deformation $E_{exp}$ and the heat $Q$ evolved during deformation:

$$E_{stor} = E_{exp} - Q \tag{1}$$

For the determination of $Q$ the crystal temperature has to be measured during the deformation process. Compared with earlier methods of deformation calorimetry, we have developed a method which is more precise in all measured quantities and uses a new procedure of evaluation of the temperature curves. This procedure of evaluation enables us to reach the accuracy of annealing calorimetry at least and to get a better accuracy than with earlier deformation calorimetry methods. The most important reasons to apply deformation calorimetry instead of annealing calorimetry are:

(1) Deformation calorimetry is suitable for analysing dynamics of plastic deformation energetically because all measured quantities are determined during the deformation process.

(2) By deformation calorimetry one can determine the change of stored energy with deformation, $e_{stor}$, for one and the same crystal along its entire deformation curve.

## 2. THE EXPERIMENT

### *Specimen*

Copper single crystals of nominal purity 99,997 (Elmore Cu of Kabelmetall, Osnabrück) were grown 4 mm in diameter and 130 mm in length by the bridgman method in a vacuum of $10^{-5}$ torr, using crucibles of highest purity graphite (EK 600 of Ringsdorff, Bad Godesberg).

### *Quantities of deformation calorimetry*

For the determination of the change of stored energy, we deform crystals by tension at constant deformation rate. Because the deformation is carried out only in very short intervals amounting to about $3 \cdot 10^{-3}$, one may write eqn (1) in terms of differences per time interval $\Delta t$:

$$\Delta E_{stor} = \Delta E_{exp} - \Delta Q \qquad (2)$$

from which the rate of energy stored in the crystal, $\dot{E}_{stor}$, is calculated:

$$\dot{E}_{stor} = \dot{E}_{exp} - \dot{Q} \qquad (3)$$

$\dot{E}_{exp}$ is calculated from the load at the crystal, $K$, and the deformation rate $\dot{L}$: $\dot{E}_{exp} = K \cdot \dot{L}$. The energy expended per time and volume unit is:

$$\dot{E}_{exp}\,(\mathrm{J\,m^{-3}\,s^{-1}}) = \sigma \cdot \dot{\varepsilon} \qquad (4)$$

$\dot{\varepsilon}$ is the relative strain rate measured directly at the crystal. The rate of heat evolved $\dot{Q}$ follows from the measured rate of temperature $\dot{T}_{pl}$ during plastic deformation and the specific heat of the crystal at constant pressure $c_p$:

$$\dot{Q}\,(\mathrm{J\,m^{-3}\,s^{-1}}) = c_p \cdot \dot{T}_{pl} \qquad (5)$$

To take into account the crystallographic orientation of the specimen axis and of the planes in which dislocation movement takes place, $\varepsilon$ and $\sigma$ are substituted by plastic shear strain $a$ and shear stress $\tau$ (for the conversion formulae see ref. 4). For this case from eqn (2) the change of stored energy with deformation, $e_{stor}\,(a)$, is calculated:

$$e_{stor}(a) = {}_{dt}\,\frac{\partial E_{stor}}{\partial a} \cong \frac{\Delta E_{stor}}{\Delta a} \qquad (6)$$

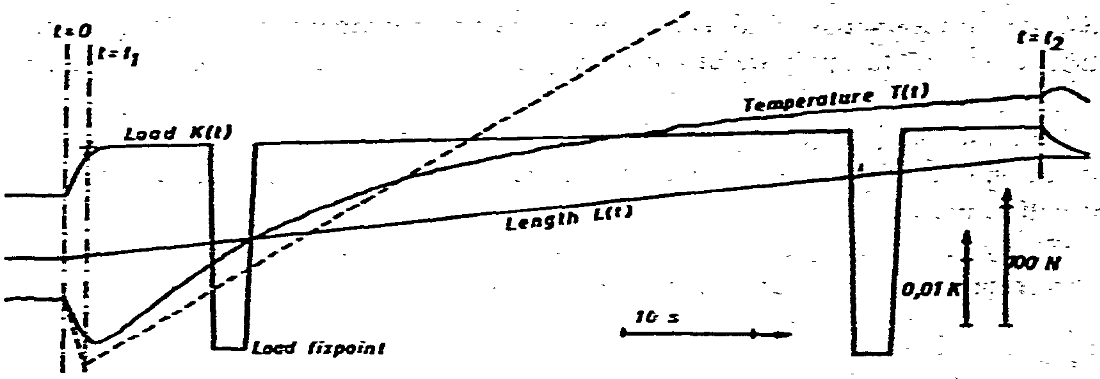


Fig. 2. Measured curves.

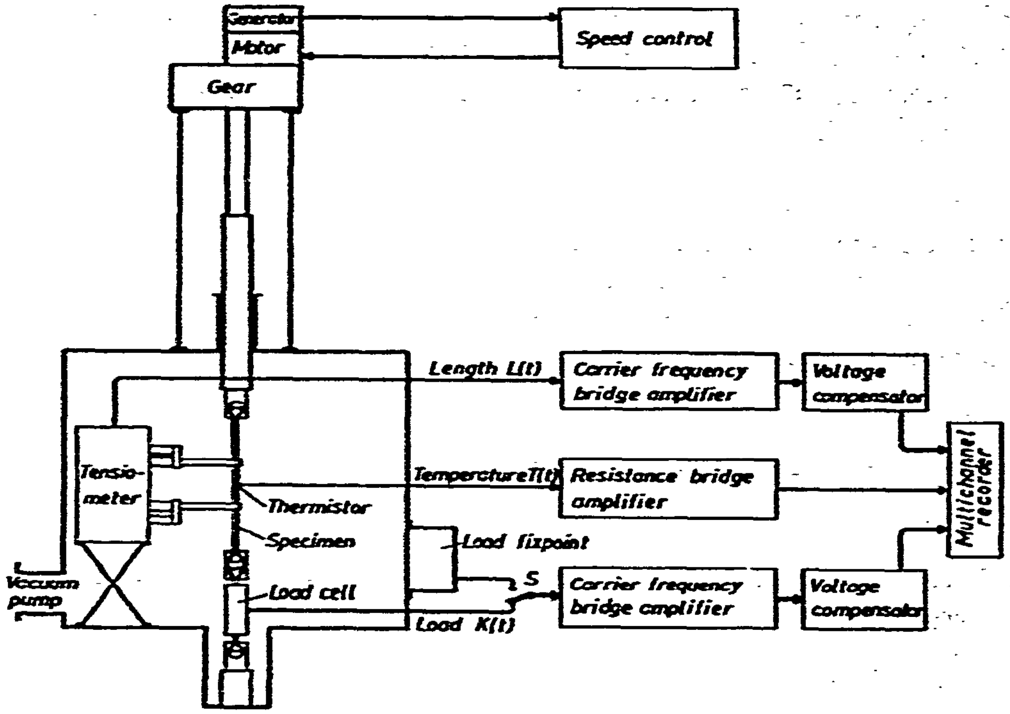


Fig. 3. Block diagram of the apparatus.

The total stored energy of the crystal, $E_{\text{stor}}$, follows by integration:

$$E_{\text{stor}}(a) = \int_{x=0}^{x=a} e_{\text{stor}}(x)\,\mathrm{d}x \tag{7}$$

*Course of a measurement*

Figure 2 shows the measured curves necessary for the measurement of the change of stored energy. Load $K(t)$, crystal length $L(t)$ and temperature $T(t)$ are recorded as a function of time $t$. For the deformation apparatus see Fig. 3. About 90%

of the load signal are electrically suppressed for reasons of evaluation accuracy. At the beginning of the experiment the crystal is subjected to an appropriate load. At $t = t_2$ the deformation is stopped and the crystal begins to relax. The temperature decrease at the beginning of the experiment is due to the thermoelastic effect. After reaching the yield point, $T(t)$ rises due to extensive plastic deformation. The temperature curve $T(t)$ is considerably affected by heat conduction. If the experiment could be carried out completely adiabatically, the specimen temperature should run along the dotted curve in Fig. 2. The large difference between the adiabatic and the real course of temperature explains the necessity of a capable evaluation procedure for the measured temperature curves.

## 3. EVALUATION OF HEAT PRODUCTION

### *The integral equation for the evaluation of the temperature curves*

Heat production in the crystal and heat conduction to the grips of the deformation machine can be described by the one-dimensional inhomogeneous equation of heat conduction:

$$\frac{\partial T(x,t)}{\partial t} - \varkappa^2 \cdot \frac{\partial^2 T(x,t)}{\partial x^2} = \frac{1}{\rho \cdot c} \cdot n(x,t) \tag{8}$$

$T$ = temperature; $x, t$ = space and time coordinates; $\varkappa^2 = \lambda/\rho \cdot c$; $\lambda, \rho, c$ are heat conductivity, density and specific heat of the crystal. $n(x, t)$ is the heat produced per time and unit volume in the specimen, in the following called power density. The measured temperature is $T(x_0, t)$, $x_0$ being the position of a thermistor which is used for temperature measurement.

We consider the case where $n(x, t)$ can be separated into factors:

$$n(x,t) = \mu(x) \cdot \mathrm{f}(t) \tag{9}$$

This means that the time-dependence of the yield of heat sources, $\mathrm{f}(t)$, is independent of their position along the crystal, the latter being described by the dimensionless function $\mu(x)$. To a rather good approximation this situation prevails during plastic (and elastic) deformation of our bar-shaped specimen, since the heat rate is controlled by the external strain rate alone, while its dependence on $x$ varies with the crystal cross-section only. In the middle region of homogeneous deformation, $\mu(x)$ is constant and may be set equal unity, arbitrarily (Fig. 4). Approaching the crystal shoulders, $\mu(x)$ vanishes, the exact course of $\mu(x)$ being unknown. Additionally, the boundary conditions of the heat conduction problem are inaccessible. Therefore, eqn (8) cannot be solved by immediate calculation. However, the evaluation of the measured $T(x_0, t)$-curves can be achieved in the following way, derived mathematically in ref. 3. It is possible to excite heat sources within the crystal of known time dependence, $\mathrm{f}(t)_{\text{test}}$. This is realized in a test measurement as will be shown below. The course of temperature of the specimen belonging to the $\mathrm{f}(t)_{\text{test}}$ in case of constant space distribution, $\mu(x)$ = constant, without any heat conduction is called test

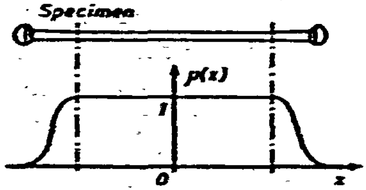


Fig. 4. Spatial distribution of power density.

function. The corresponding true course of temperature, measured at the position $x_0$, is the response function $T(x_0, t)_{\text{test}} \propto h(t)$ of the heat conduction system. The course of $h(t)$ is determined by the impressed time function of heat production $\mathrm{f}(t)_{\text{test}}$ and the result of the boundary conditions of heat conduction and space distribution $\mu(x)$ underlying the experiment. With the aid of this function the solution of eqn (8) can be written for *any* $\mathrm{f}(t)$, but under the same $\mu(x)$ and the same boundary conditions, in the form of integral equations[3]:

$$T(x_0, t) = \frac{1}{\rho \cdot c} \cdot \int_{t' \to -\infty}^{t'=t} \frac{\partial \mathrm{f}(t')}{\partial t'} \cdot h(t - t') \cdot \mathrm{d}t' \tag{10}$$

$$T(x_0, t) = -\frac{1}{\rho \cdot c} \cdot \int_{t' \to -\infty}^{t'=t} \mathrm{f}(t') \cdot \frac{\partial h(t - t')}{\partial t'} \cdot \mathrm{d}t' \tag{11}$$

As $h(t)$ is already included in the integration over $x$, this variable does not appear in eqns (10) and (11).

To realize experimentally the response function of the known heat rate $\mathrm{f}(t)_{\text{test}}$, we perform a pure elastic deformation of our specimen at constant stress rate, $\dot{\sigma}_{\text{test}}$. The description of the time function $\mathrm{f}(t)_{\text{test}}$ corresponding to the externally impressed stress rate is straightforward. We only have to describe the adiabatic homogeneous case, since $\mathrm{f}(t)$ is independent of the heat conduction and spatial inhomogeneities. First, we use the equation of thermoelastic effect for adiabatic homogeneous deformation:

$$\dot{T}_{\text{test}}^{\text{ad}} = -\frac{\alpha \cdot T_{\text{abs}}}{c_p} \cdot \dot{\sigma}_{\text{test}} = E \cdot \dot{\sigma}_{\text{test}} \tag{12}$$

(uniaxial tensile deformation, constant stress rate $\dot{\sigma}_{\text{test}}$). This equation is valid not only for isotropic materials but also for elastically anisotropic cubic crystals. $\alpha$, $T_{\text{abs}}$, and $c_p$ are linear extension coefficient, absolute temperature, and specific heat at constant pressure, respectively. Second, we derive the connection of $\mathrm{f}(t)$ with the temperature change at the position of the thermistor, $T(x_0, t)$: In case of homogeneous adiabatic conditions, the equation of heat conduction (8) yields:

$$\frac{\partial T(x_0, t)}{\partial t} = \frac{1}{\rho \cdot c} \cdot \mathrm{f}(t) \tag{13}$$

with $\mu(x_0)$ normalized to unity, as introduced above. To apply eqn (10), one has to guarantee that $f_{test}\,(t < 0) = 0$, thus we write:

$$f(t)_{test} = \rho \cdot c \cdot \dot{T}^{ad}_{test}\,\theta(t) \tag{14}$$

using the stepfunction $\theta(t)$, ($\theta(t < 0) = 0$, $\theta(t \geqslant 0) = 1$). The time derivative of (14) becomes:

$$\dot{f}(t)_{test} = \rho \cdot c \cdot E \cdot \dot{\sigma}_{test} \cdot \delta(t) \tag{14a}$$

with $\delta(t)$ = Dirac function. The $\delta$-function makes the integration of eqn (10) very simple:

$$T(x_0, t)_{test} = \frac{1}{\rho \cdot c} \cdot \int\limits_{t' \to -\infty}^{t'=t} \rho c \cdot E \cdot \dot{\sigma}_{test} \cdot \delta(t') \cdot h(t - t') \cdot \mathrm{d}t'$$

$$T(x_0, t)_{test} = E \cdot \dot{\sigma}_{test} \cdot \mathrm{h}(t) \tag{15}$$

differing from zero for $t > 0$.

$T(x_0, t)_{test}$ is the measurable variation of temperature due to the thermoelastic experiment (Fig. 5). With $T(x_0, t)_{test}$, also $h(t)$ is known via eqn (15). Supposing that the space distribution of heat production, $\mu(x)$, is the same during plastic and elastic deformation to a good approximation, the temperature variation due to plastic deformation, $T(x_0, t)$, will be shown to be represented in terms of $h(t)$. As $f(t)_{test}$ is fully determined, $T(x_0, t)$ is also calibrated.

### *Solution of the integral equation by means of a parameter representation*

The numerical calculation of the integral kernel $f(t)$ (respectively $\partial f/\partial t$) for given temperature curves $T(x_0, t)$ and $h(t)$ is difficult. In general, a strongly oscillating function $f(t)$ results since $T(t)$ and $h(t)$ are affected by noise. This behaviour of such

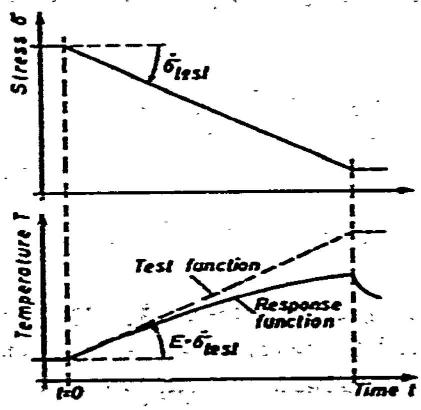


Fig. 5. Test and response function.

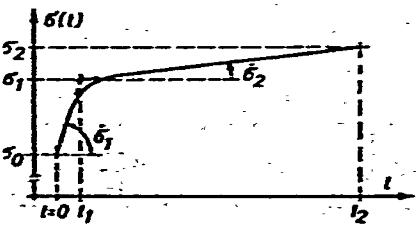


Fig. 6. The course of stress.

integral equations is known[5, 6]. As an alternative to their general solution we start from a parameter representation of f($t$). The introduced parameters are calculated using least squares analysis. The parameter representation is based on the experimentally given situation. As the crystal deforms plastically and elastically during an experiment, the heat rate of the specimen consists of two parts:

$$f(t) = f(t)_{\mathrm{pla}} + f(t)_{\mathrm{thermo}}$$

or equivalently:

$$\dot{T}^{\mathrm{ad}}(x_0, t) = \dot{T}^{\mathrm{ad}}_{\mathrm{pla}}(x_0, t) + \dot{T}^{\mathrm{ad}}_{\mathrm{thermo}}(x_0, t)$$

The adiabatic plastic and thermoelastic temperature changes are easily derived from the course of stress $\sigma(t)$. Because the deformation is performed at constant rate, the heat production during plastic deformation is written proportional to the course of stress, $\sigma(t)$. Thus, we get for $t \geqslant t_1$:

$$\dot{T}^{\mathrm{ad}}_{\mathrm{pla}} = b \cdot [\sigma_1 + \dot{\sigma}_2 \cdot (t - t_1)] \cdot \theta(t - t_1)$$

$b$ and $t_1$ are unknown parameters. For the course of stress and the meaning of $\sigma_1$, $\dot{\sigma}_2$ see Fig. 6.

The thermoelastic change of temperature results from the stress rate, $\dot{\sigma}(t)$. From eqn (12) we get:

$$\dot{T}^{\mathrm{ad}}_{\mathrm{thermo}}(x_0, t) = E \cdot \{\dot{\sigma}_1 \cdot [\theta(t) - \theta(t - t_1)] + \dot{\sigma}_2 \cdot \theta(t - t_1)\}$$

Again $\theta$-functions are introduced to guarantee that the slope $\dot{\sigma}_1$ applies for $0 \leqslant t \leqslant t_1$ whereas $\dot{\sigma}_2$ is effective for $t \geqslant t_1$. The only simplification consists in representing $\sigma(t)$ by two straight lines. Since the crystal is never deloaded between two measurements, yield point phenomena are negligible. Thus, the straight lines are good approximations of $\sigma(t)$ except for times close to $t_1$ (Fig. 2).

For the application of the integral equation (10) $\dot{T}^{\mathrm{ad}}(x_0, t)$ is multiplied by $\rho \cdot c$ and differentiated yielding $\partial f/\partial t$. The subsequent integration is nearly as simple as in the previous case:

$$T_{\mathrm{param}}(x_0, t) = [b \cdot \sigma_1 + E \cdot \dot{\sigma}_2] \cdot h(t - t_1) + E \cdot \dot{\sigma}_1 \cdot [h(t) - h(t - t_1)] + b \cdot \dot{\sigma}_2 \cdot \int_{u=0}^{u=t-t_1} h(u) \cdot du \qquad (16)$$

Using least squares analysis the parameters $b$ and $t_1$ are determined by making $T_{\mathrm{param}}(x_0, t)$ the optimal approximation of the measured temperature curve $T(x_0, t)$. From $b$ the temperature rate of plastic deformation, which is the aim of the evaluation, can be calculated:

$$\dot{T}^{\mathrm{ad}}_{\mathrm{pla}}(x_0, t = t_1) = b \cdot \sigma_1 \qquad (17)$$

## 4. EXPERIMENTAL DETAILS

### *The deformation machine*

Figure 3 shows the block diagram of the whole apparatus. In the deformation machine, bar-shaped crystals can be deformed under tension. Crystal and load cell are located in a vacuum chamber to suppress heat convection. We succeeded in designing a machine of rather high stiffness. The spring constant of the whole apparatus, including load cell and all necessary joint elements of a real measurement, amounts to $10^4$ N $mm^{-1}$. This equals the stiffness of the crosshead of an Instron deformation machine (floor model TT-BM-L) without load cell and joint elements.

### *Measurement of load*

Systematic errors of load measurement result from the temperature drift of the bridge amplifier and from changes in cable capacitance. By means of a reference signal, named load-fixpoint, the influence of such disturbances could be eliminated. For this purpose, the strain gauges of the load cell are operated as a half-bridge. A further half-bridge consisting of wire resistances is connected in parallel (Fig. 7). With switch S, it is possible to change from load-cell to load-fixpoint. Since all disturbances outside the unit consisting of load-cell and reference half-bridge cause the same error signal, these influences are eliminated if the load is measured from the load-fixpoint. Using this fixpoint we gain at least one order of magnitude in accuracy of load measurement, mainly long time drift is suppressed in this way.

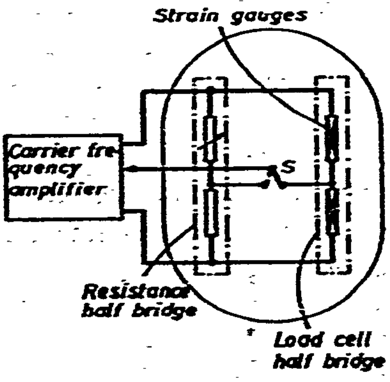


Fig. 7. Load and fixpoint circuit.

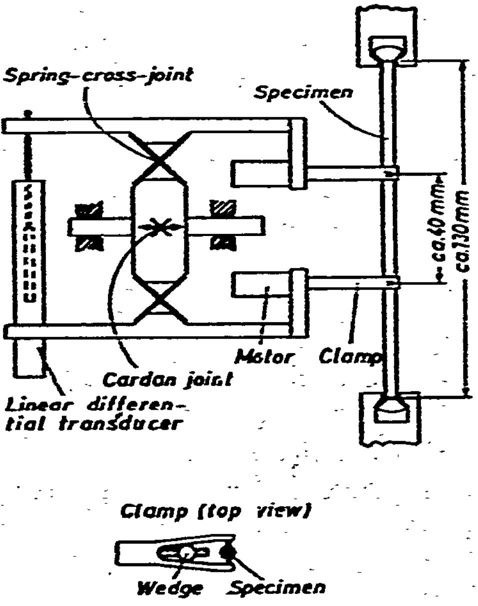
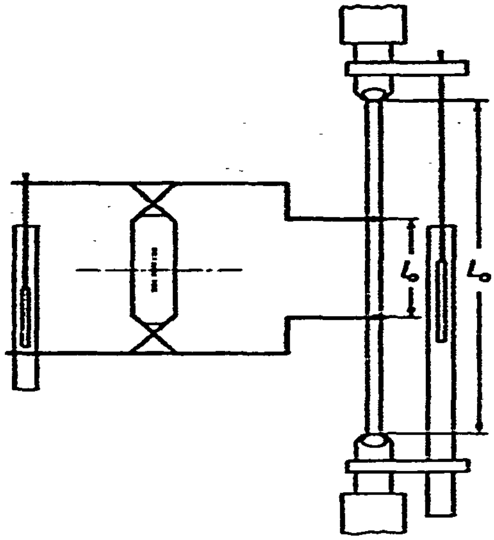


Fig. 8. The tensiometer.



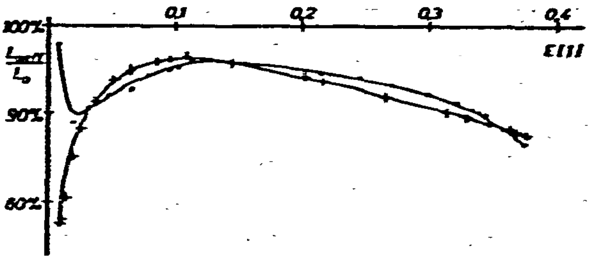


Fig. 9. Comparison with externally measured strain.

Fig. 10. Course of the effective crystal length of two single crystals oriented for single glide.

*Measurement of deformation rate*

The determination of the deformation rate (resp. strain), which is effective in the middle part of the crystal, suffers from large uncertainties. The elasticity of the deformation machine and the influence of the grips cause a loss of external deformation velocity of about 10% (though our machine is a rather hard one). To avoid such errors, we measure the deformation rate directly at the crystal by means of an appropriate tensiometer. The tensiometer is given schematically in Fig. 8. Its essential features are:

(1) Near its centre of mass, it is suspended cardanically and free in translation perpendicular to the crystal.

(2) Two cross-joints transmit the elongation towards a linear differential transducer. These spring joints are free of mechanical play.

(3) For attachment to the crystal two clamps are forced apart by micro-motors. After deloading of these clamps the crystal is held elastically.

For comparison with externally measured strain, we used the experiment shown in Fig. 9. To get quantitative results, we call $\Delta L_{ext}$ the elongation measured at the grips, $\Delta l$ the elongation measured directly at the crystal. $L_0$ and $l_0$ denote the corresponding reference lengths. The external and true strain are given by:

$$\varepsilon_{ext} = \frac{\Delta L_{ext}}{L_0}; \varepsilon = \frac{\Delta l}{l_0}$$

To get a measure for the deviations between $\varepsilon_{ext}$ and $\varepsilon$, it seems convenient to define an effective crystal length $L_{0\,eff}$ (as is often done in literature):

$$\frac{\varepsilon - \varepsilon_{ext}}{\varepsilon} = \frac{L_0 - L_{0\,eff}}{L_0}$$

If $L_{0\,eff}$ would turn out to be constant $\varepsilon$ could be calculated from the externally measured elongation $\Delta L_{ext}$, using $\varepsilon = \Delta L_{ext}/L_{0\,eff}$. However, Fig. 10 shows that there exists no constant effective crystal length. Represented is the ratio $L_{0\,eff}/L_0$ [%] as a function of relative strain for two copper crystals oriented for single glide. These results can be explained as follows: Near the grips, the crystal deforms less than in its middle part. The deviations are most pronounced at the beginning of the deformation. They are not reproducible in the low deformation region, since the crystal has to adjust to the grips.

Because the stored energy itself amounts only to 10% of the expended work, the measurement of deformation rate must necessarily be performed directly at the crystal. This holds for all deformation calorimetry methods. Otherwise systematic errors of 100% may arise in the determination of stored energy.

*Measurement of temperature*

To determine the heat evolved during plastic deformation, the temperature is measured by means of ntc-resistors (Siemens K19). They consist of a small glazed semiconductor bead, about 0,4 mm in diameter with Mo-leads of diameter 20 $\mu$m. To measure the temperature, the resistance of the thermistor being attached to the crystal is determined with the aid of a Wheatstone dc-bridge containing a chopper stabilized pre-amplifier.

Technology of establishing thermal contact between thermistor and crystal. The main problem in establishing thermal contact between crystal and thermistor arises from the deformation of our specimen. This deformation can reach 50% for a tensile deformed single crystal. Our method of attaching thermistors to the crystal is illustrated in Fig. 11. First, the lead wires are glued near the resistor bead by an adhesive (UHU Universal) which does not harden completely and therefore adheres to the crystal even at large plastic strain. Second, the thermistor is wholly embedded in Silicon paste (Wacker Chemie, München; paste K12). It has vanishing electrical conductivity. Because of its low viscosity (at room temperature) the thermal contact remains uneffected by plastic deformation. An estimate of the heat capacity $C_{total}$ of thermistor and heat conduction paste amounts to $C_{total} = 5 \cdot 10^{-4}\,\mathrm{J\,K^{-1}}$. This equals the heat capacity of about $1.5 \cdot 10^{-3}$ g Cu. Because our crystals have a mass of 15 g

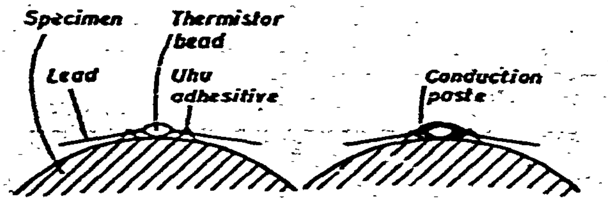


Fig. 11. Technology of establishing thermal contact between crystal and thermistor.

and are very good heat conductors, the feed back of our temperature element on the crystal temperature itself remains negligible.

## 5. RESULTS

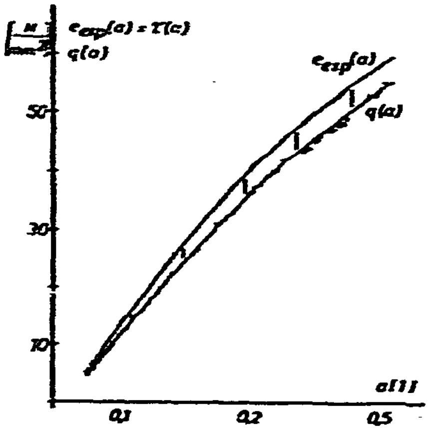
Figures 12–14 show results of measurements on a copper crystal oriented for single glide, deformed at room temperature. From the measured energies per time unit $\dot{E}_{exp}$, $\dot{Q}$, $\dot{E}_{stor}$, the respective energy changes with deformation are calculated: We generalize definition (6) and make use of the condition $\dot{a}$, $T$ = constant, given in the experiment, to get:

$$\begin{aligned} e_{stor}(a) &= \frac{\partial E_{stor}}{\partial a} = \frac{\dot{E}_{stor}}{\dot{a}} \\ e_{exp}(a) &= \frac{\partial E_{exp}}{\partial a} = \frac{\dot{E}_{exp}}{\dot{a}} = \frac{\tau \cdot \dot{a}}{\dot{a}} = \tau(a) \\ q(a) &= \frac{\partial Q}{\partial a} = \frac{\dot{Q}}{\dot{a}} \end{aligned} \tag{18}$$

$e_{exp}(a)$, $q(a)$ and $e_{stor}(a)$ are represented as functions of mean shear strain $a$ in Figs. 12 and 13. The use of $e_{exp}(a)$, $q(a)$ and $e_{stor}(a)$ instead of the measured energies per time $\dot{E}_{exp}$, $\dot{Q}$ and $\dot{E}_{stor}$ has two advantages:

(1) The trivial proportionality of energies per time to the deformation rate is separated. The latter statement, however, does not mean that $e_{exp}$, $q$ and $e_{stor}$ are independent of strain rate $\dot{a}$.

(2) $e_{exp}(a)$ turns out to be identical with the resolved shear stress $\tau(a)$, thus also $q(a)$ and $e_{stor}(a)$ become related to this well-known physical quantity, $q(a)$ and $e_{stor}(a)$ are stresses, too.



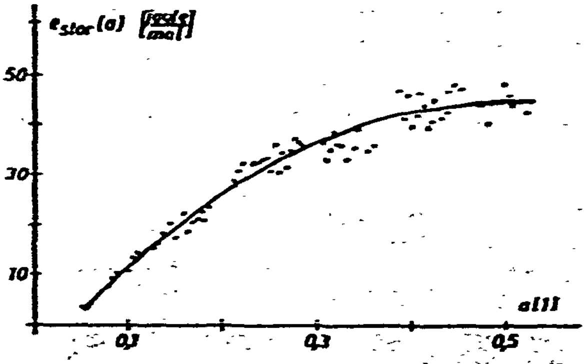


Fig. 12. Shear stress $\tau(a)$ and change of heat evolved $q(a)$.

Fig. 13. Change of stored energy with shear strain $e_{stor}(a)$.

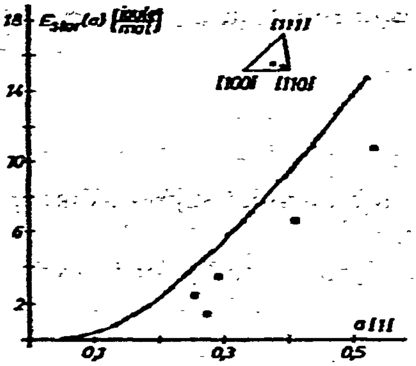


Fig. 14. Stored energy $E_{stor}$; large symbols mark results of Gottstein (1975).

79 measurements were performed, divided into 6 sequences. In Fig. 12 the division in sequences is marked by short vertical lines. Obviously, the sequences join each other. Because every sequence is wholly independent of the other (two sequences mean two independent temperature calibrations, two independent tensiometer attachments and so on), the good fit of successive sequences demonstrates the reproducibility of our measurements. Figure 14 shows the total stored energy, $E_{stor}$, calculated by summation of the measured changes of stored energy, $e_{stor}(a)$, see eqn (7). For comparison with annealing calorimetry, measurements at comparable single crystals of Gottstein[7] are also given. Obviously, our accuracy of determining stored energies is better than that of annealing calorimetry. One of the main reasons may lie in the fact that our measurements were gained at one and the same crystal whereas every value of stored energy determined by annealing necessarily belongs to another specimen.

The results of measurement of stored energies by different authors using various apparatus differ appreciably. In particular, the use of different methods, annealing and deformation calorimetry, led to quite distinct results. Representative for these discrepancies is a comparison of the numerous deformation calorimetry results by Wolfenden[8, 9] and results of annealing calorimetry[10]. The stored energies of polycrystals measured by Wolfenden[8] are larger, up to a factor 10, than the results of Steffen[10]. A convincing physical explanation for such a large difference has never been given, systematic experimental errors excluded. The order of magnitude of the results of Steffen and Gottstein was verified by a comparative measurement[11] using different annealing calorimeters but specimen which were cut from one and the same (heavily) deformed crystal. Figure 14 shows that our stored energies and those of Gottstein are of the same order of magnitude. By this the large discrepancies between annealing and deformation calorimetry seem to be overcome. As to the deformation calorimetry measurements of Wolfenden, possibly they fail in the exact evaluation of measured temperature curves. Wolfenden seems neither to evaluate the whole curve (he uses a certain maximum temperature), nor to give a definitive method to take into account the heat conduction.

The physical discussion of these results together with results at a [100]-crystal will be published in ref. 12.

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