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INVESTIGATIONS OF INTERNAL FRICTION AND CONSTANTS OF ELASTICITY OF SOLIDS BY THE ACOUSTIC RESONANCE METHOD

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Results of measurements of the internal friction, Poisson's constant and Young's modulus for various metals: copper, aluminium, copper without oxygen, brass, aluminium and silicon bronzes, in different states of deformation and heat treatment, were presented. Discovery of the formerly unknown anomaly of internal friction in a plastically deformed silicon bronze BK31, occurring in the temperature range 400—500 K, was discussed. A number of effects that accompany this phenomenon were described, together with attempts of explanation of its physical mechanism.

1. Introduction

Investigations of the internal friction (IF) of solids have been carried out in the world science with great intensity, since they enable us to study the structure of the matter and, especially, to establish the role played by the defects of the crystal lattice in forming specific features and properties of various materials, metals in particular. Generation or elimination of proper defects or changing possibilities of their interactions with other kinds of defects (for instance, by adding appropriate chemical elements) enable us to draw important conclusions.

Equipment for investigations of dislocation structures is very complicated and expensive. However, it is possible to investigate the dislocations indirectly, by using measurements of the constants of elasticity (CE) and internal friction as functions of the temperature. Investigations of different metals, carried out at the Department of Acoustic Physics (DAF) of the IFTR PAS, have led us to the discovery of non-typical changes of IF for silicon bronze BK31 [1—3]. This bronze has a property which characteristic for it and has not been found in other metals, i.e. a significant (tenfold) decrease of IF, which occurs in the temperature range 400—500 K, in the state of plastic deformation. Discovery of this phenomenon has caused our interest in its reasons. Thus, the main purpose of the following investigations was to study the mechanisms causing this phenomenon. The way leading to that consists in geting information on the submicroscopic processes that occur in BK31 bronze and cause the mentioned macroscopic effects connected with IF under the specified conditions. This phenomenon was not known in the world science before, and was discovered by us and announced at two international conferences [2, 4]. At the DAF, the investigations were carried out by applying the resonance elastometer, i.e. the apparatus for measurements of IF by means of the acoustic resonance method [6, 7]. Other investigations performed by means of a torsional pendulum and a quartz resonance oscillator were made in cooperation with Gdańsk University of Technology and Physical-Technological Institute in St. Petersburg, respectively.

2. Conditions of investigations and results

Generally, the phenomenon of friction and the energy dissipation connected with it can be external, for instance friction of surfaces sliding in contact with one another, or internal, for instance, displacements of atoms pressed against each other, motion of dislocations, interactions of magnetic and electric fields in ferromagnetic and dielectric materials.

Changes of IF are most frequently recorded as functions of different parameters, such as the amplitude of deformations of a sample, the frequency of deformation and the temperature of a sample. Their analysis can give a lot of information on the internal processes induced by mobility of defects of the crystal lattice and their different transformations.

Changes of the modulus of elasticity E and Poisson's constant ν are usually measured together with measurements of IF as functions of the same variables. In general, important changes or anomalies do not occur during measurements of these parameters; namely, when temperature increases, Poisson's constant ν also increases monotonically and Young's modulus E decreases. The changes of IF cause the deflection of the curve $E(T)$. This deflection is easy to notice due to the measurements made by means of a torsional pendulum, and slightly less visible when the resonance elastometer is applied.

2.1. The dependence of IF *on the amplitude of deformation*

The measurements of IF can be done in a wide range of amplitudes of deformation. The applicable amplitudes depend on the frequency and the method used in measurements.

In the case of high frequencies, $10^5 - 10^{10}$Hz, the measurements of IF can be done in the range of the amplitudes of relative deformation of about $10^{-9} - 10^{-8}$, which are too small to cause a permanent deformation of the crystal structure. The frequencies $10^{-2} - 10^4$ Hz and the amplitude $10^{-3} - 10^{-2}$ that can distrub the physical cohesion of the crystals are the second extreme case. In measurements of IF by the torsional pendulum and resonance elastometer, the amplitude of relative deformation is within the ranges $10^{-6} - 10^{-3}$ and $10^{-9} - 10^{-8}$, respectively.

In the first approximation one can notice that, in the range of low deformations, the value of IF is independent of the amplitude. On the other hand, for higher deformation, this value increases when the amplitude increases (Fig. 1) [16]. This fact indicates the existence of two different mechanisms of energy dissipation that occur in the ranges of the relatively low and high amplitudes. The limit between these ranges corresponds to the points of inflexion of the curves presented in Fig. 1. The value of the amplitude that starts the mechanism of the internal friction dependent on the amplitude is different for various materials and, for the same material, depends on the temperature and frequency. This fact can be explained when the influence of temperature and frequency on the mobility of lattice defects is considered.

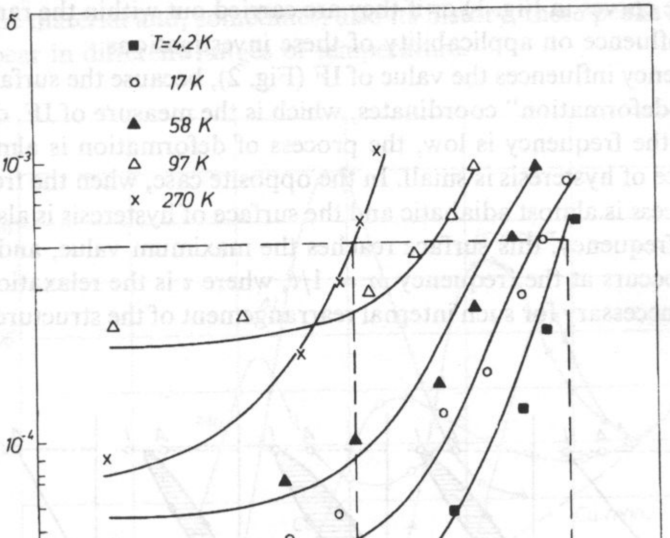

Fig. 1. Dependence of internal friction on the amplitude.

Generally, the total value of IF, Q^{-1}, consists of two parts,

$$Q^{-1} = Q_0^{-1} + Q_z^{-1}, \tag{2.1}$$

where $Q^{-1} = Q_0^{-1}$ is the component independent of the amplitude and occurs at lower amplitudes up to the point of inflexion, Q_z^{-1} is the component for higher amplitudes and depends on the amplitude.

2.2. The dependence of IF *on the frequency*

By applying different measurement methods, it is possible to investigate IF in the very range of frequencies — from $10^{-4}... 10^{-3}$ Hz to $10^{11}... 10^{12}$ Hz. Investigation made by means of the torsion pendulum and the resonance elastometer can be done in the ranges $10^{-2}... 10^{2}$ Hz and $5.10^{3}... 3.10^{4}$ Hz, respectively.

The known results of investigations of the IF dependence on the frequency are inconsistent with each other, not only in the case of different materials, but even for the same material.

The fact, whether the investigations are made at a sufficiently low amplitude of deformation, for which $Q^{-1} = Q_0^{-1}$ is still independent of the amplitude (the horizontal sections of the curves in Fig. 1) or if they are carried out within the range of high Q_z^{-1}, has a great influence on applicability of these investigations.

The frequency influences the value of IF (Fig. 2), because the surface of hysteresis in the „stress-deformation" coordinates, which is the measure of IF, depends on this frequency. If the frequency is low, the process of deformation is almost isothermal and the surface of hysteresis is small. In the opposite case, when the frequency is very high, this process is almost adiabatic and the surface of hysteresis is also small. At the intermediate frequency, this surface reaches the maximum value, and then IF is the largest. This occurs at the frequency $\omega = 1/\tau$, where τ is the relaxation time, i.e. the time which is necessary for such internal rearrangement of the structure of a body that

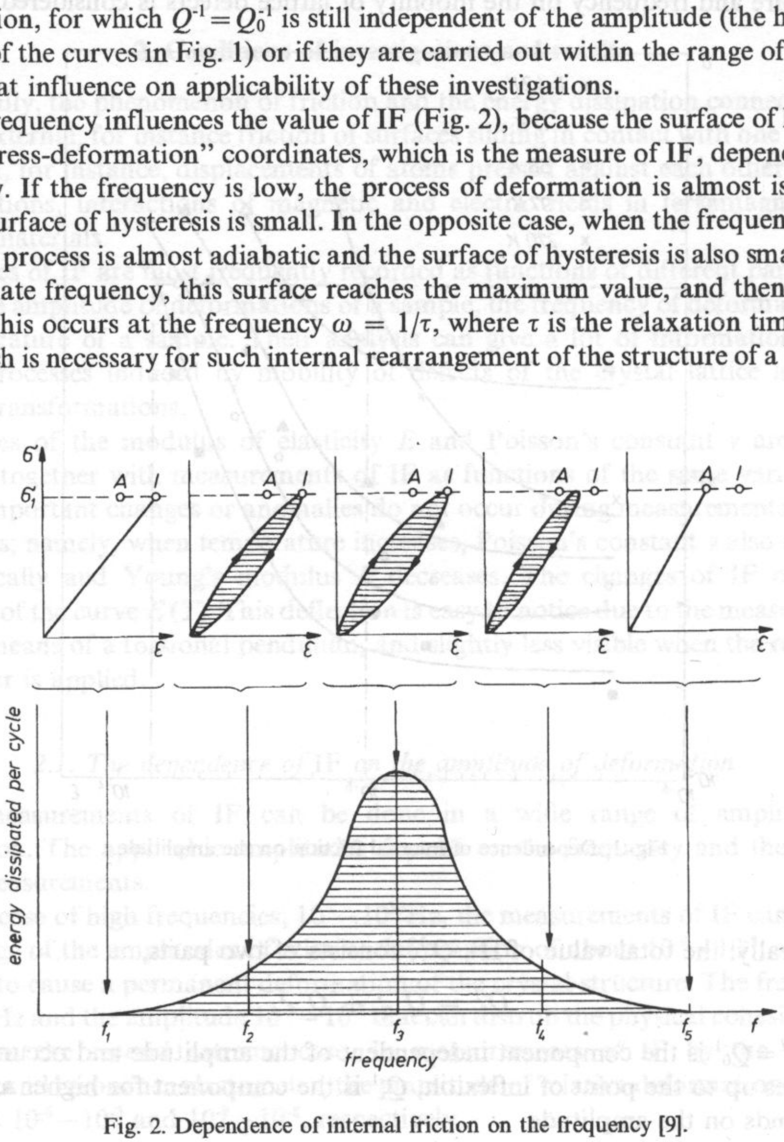

Fig. 2. Dependence of internal friction on the frequency [9].

the deformation of this body changes by $1/e$ of its initial values, where e is the base of natural logarithms.

2.3. The dependence of IF on the temperature

The investigations of most of the materials indicate that, in the range of temperature from 4 K to 0.5—0.6 T_t, where T_t is the melting temperature, the value of $Q^{-1}(T)$ increases monotonically almost lineary when T increases, whereas, at higher temperature, the increase of $Q^{-1}(T)$ is more rapid-exponential (Fig. 3). In many cases, despite this kind of changes, the peaks appear on the curves of $Q^{-1}(T)$, which are caused by the specific influence of different kinds of the crystal lattice defects. According to the material and, sometimes, also its history, these peaks have different height and appear in different ranges of temperature.

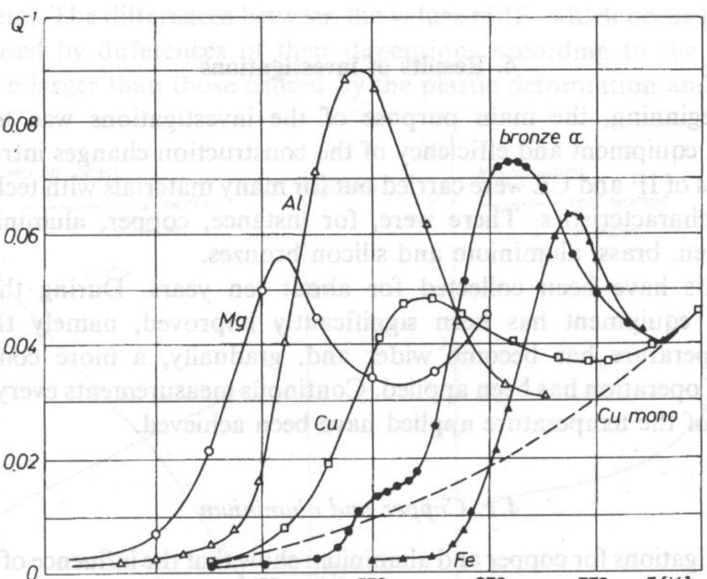

Fig. 3. Typical changes of internal friction as a function of temperature.

3. The samples and procedure of measurements

In the measurements made at the DAP by means of the resonance elastometer, the samples in the form of disks with diameter 30 mm and thickness 1 — 3 mm were used. They were supported at three points placed symmetrically with respect to the center, and in line with the nodes of bending vibrations for two different modes. The electronic setup excited the sample to oscillations in the first or second modes with two different resonance frequencies, which depend on the temperature. Knowing the values of these frequencies, the dimensions of the sample and the density of the material, the CE have been calculated. The IF has been determined by the measurements of the amplitude of vanishing free oscillations of the sample excited to the resonance vibrations [6, 7].

To achieve a low temperature, the measurement cell together with the sample were submerged in a liquid nitrogen and this caused cooling of the sample to about –160° (about 110 K) after a few hours. Total or partial emerging of the cell from the liquid nitrogen made the temperature increase faster or slower. The electric heater was used to achieve higher temperature. The rate of the temperature increase was regulated by changing the power of the heater. Construction of the experimental equipment and/or the elastic properties of the tested materials limit the maximal applied temperature.

The operating principle of the torsion pendulum has been described, for instance, in [15].

4. Results of investigations

In the beginning, the main purpose of the investigations was to verify the measurement equipment and efficiency of the construction changes introduced. The measurements of IF and CE were carried out for many materials with technical purity and known characteristics. There were, for instance, copper, aluminium, copper without oxygen, brass, aluminium and silicon bronzes.

The results have been collected for about ten years. During that time the measurement equipment has been significantly improved, namely the range of possible temperature has become wider and, gradually, a more complete automatization of operation has been applied. Continous measurements every 1 deg in the whole range of the temperature applied have been achieved.

4.1. Copper and aluminium

The investigations for copper and aluminium show that the influence of temperature on CE is typical; when the temperature increases, then $E(T)$ decreases monotonically and $\nu(T)$ increases. The changes of $Q^{-1}(T)$ are similar to those presented in Fig. 3, which is a typical diagram of the monotonic increase of IF as a function of temperature.

4.2. Copper without oxygen of the MOB type

Besides the general testing properties of copper without oxygen, the special purpose of our measurements is to verify whether, after application of high hydrostatic pressures, the changes in the elastic properties of metal occur, like after the mechanical plastic deformation. CE was measured in the range of temperature 20 ... 230°C, with the step of 10 deg. IF was investigated for the temperature 30, 100, 170 and 230°C.

Results of these measurements show that the influence of hydrostatic pressure is completely similar to that of the mechanical deformation, despite the fact that the shape of the samples was practically unchanged when pressures of 300, 450 and 600 MPa were used. This result is rather unexpected, since it should not occur theoretically.

4.3. *Brass* MO58

Brass MO58 was investigated at the temperature from about 110 to 800 K, after a single plastic deformation (near 9%) and annealing. Typical functions $Q^{-1}(T)$, $E(T)$ and $\nu(T)$ have been found; when the temperature increases, the values of $E(T)$ and $\nu(T)$ change almost linearly, and $Q^{-1}(T)$ increases — slowly in the beginning Fig. 4, and faster above the temperature 420 K, like in Fig. 3. The value of IF, in the whole range of temperature, does not differ significantly for the plastically deformed and annealed metal. The differences between the values of IF, which occur in the samples and are caused by differences of their dimensions according to the technological tolerance, are larger than those caused by the plastic deformation and annealing.

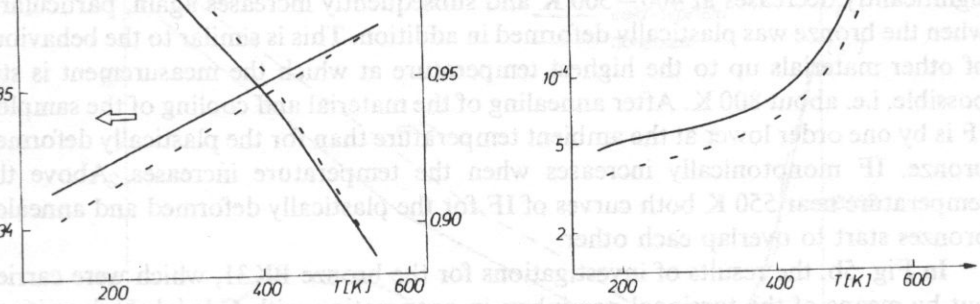

Fig. 4. Constants of elasticity and internal friction for brass as a function of the temperature.

4.4. *Aluminium bronze BA1044*

The samples of aluminium bronze, which were out from a raw rod, were heated during the measurements to the temperature at which IF could be measured. Monotonic increase of $Q^{-1}(T)$ and typical changes of CE were obtained.

Similarity of the changes of $Q^{-1}(T)$ is very important in the case of materials described since, on this basis, a different behavior of $Q^{-1}(T)$ for the silicon bronze becomes very significant.

4.5. Silicon bronze BK31

The investigations made at the DAP of IFTR have led us to the discovery of non-typical changes of $Q^{-1}(T)$ for silicon bronze BK31 [1–3]. For this bronze, a significant about tenfold decrease of IF occurs in the plastically deformed state at the temperature from 400 to 500 K (Fig. 5a). Consequently, this bronze has been tested under various conditions and by means of other methods. Previous investigations were done in the whole range of temperature, i.e. from about 110 to 800 K. Later, this range was limited to the temperature above 300 K, since the anomalies of the properties occur at the temperature in the range 400 – 500 K.

Four types of rods made of bronze BK31 were used to cut out the samples. First rod was simply a raw material from metallurgical works, the second and third ones were, in addition, axially compressed by about 4.5% and 15%, respectively. The fourth one was annealed for one hour at the temperature 780 K and subsequently axially compressed by about 15%. Increasing temperature was applied to the sample made of these rods (Fig. 5 a). Some of these samples, after annealing during the measurements, were additionally exposed to the action of a hydrostatic pressure and measured again. The results are similar to those obtained after application of the mechanical deformation.

For the BK31, after the initial increase at the temperature 110 – 300 K, $Q^{-1}(T)$ significantly decreases at 400 – 500 K and subsequently increases again, particularly when the bronze was plastically deformed in addition. This is similar to the behaviour of other materials up to the highest temperature at which the measurement is still possible, i.e. about 800 K. After annealing of the material and cooling of the samples, IF is by one order lower at the ambient temperature than for the plastically deformed bronze. IF monotonically increases when the temperature increases. Above the temperature near 550 K both curves of IF for the plastically deformed and annealed bronzes start to overlap each other.

In Fig. 5b, the results of investigations for the bronze BK31, which were carried out by means of the torsional pendulum in cooperation with Gdańsk University of Technology, are shown. A complete compliance of results follows from the comparison of Figs. 5a and 5b.

5. The most important results for the silicon bronze

Trying to explain the abnormal changes of IF for the silicon bronze, the investigations of metallographic specimens was made before and after annealing. They indicate that the grains of the metal become smaller during annealing. To check whether this fact of reduction of the grain dimensions is the reason of the anomaly of

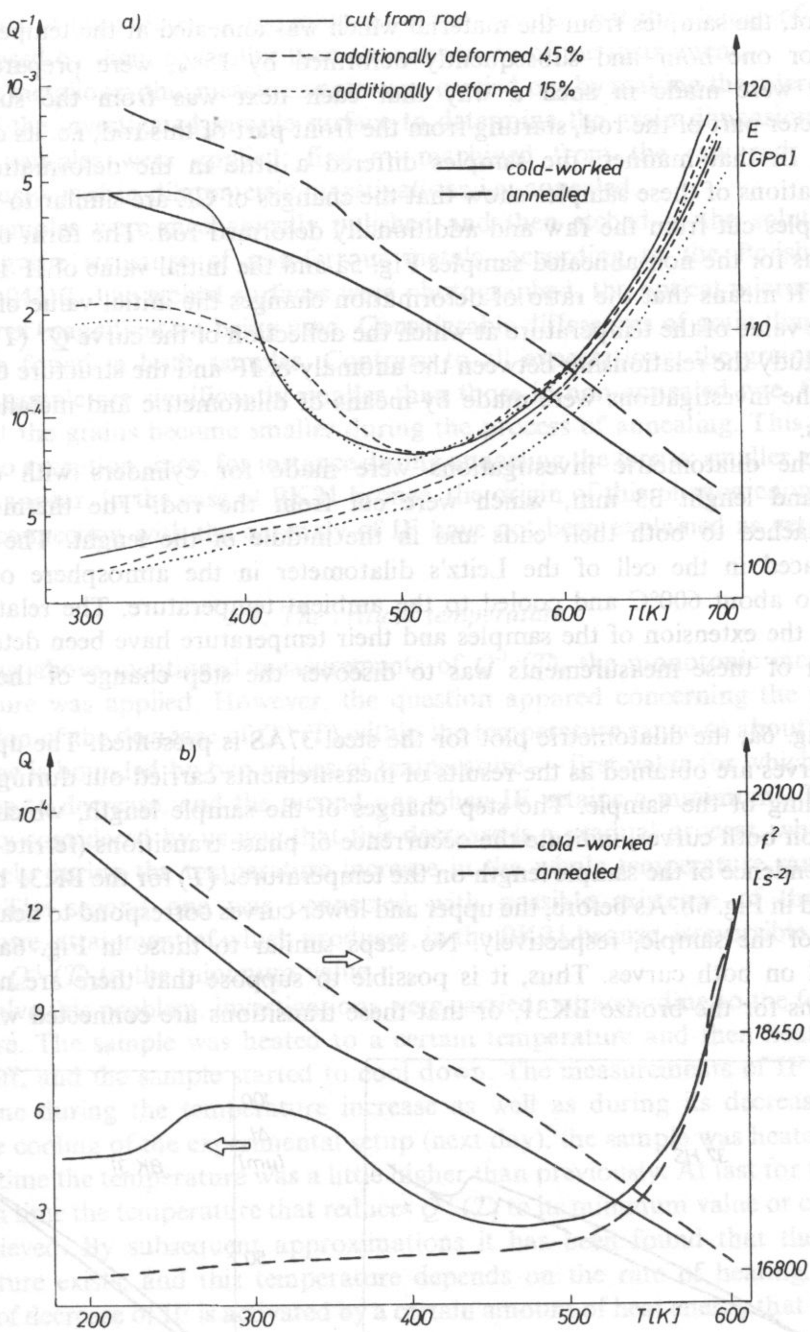

Fig. 5. Internal friction and Young's modulus of the plastically deformed and annealed bronze BK31 measured by means of: a) — resonance elastometer, b) — torsional pendulum.

IF or not, the samples from the material which was annealed at the temperature of 770K for one hour and subsequently deformed by 15%, were prepared. Four samples were made in such a way that each next was from the subsequent 6-milimeter part of the rod, starting from the front part of this rod, i.e. its deformed surface. In that manner, the samples differed a little in the deformation ratio. Investigations of these samples show that the changes of CE are similar to those for the samples cut from the raw and additionally deformed rod. The form of Q^{-1} (T) is such as for the non-annealed samples Fig. 5a and the initial value of IF is slightly greater. It means that the ratio of deformation changes the initial value of IF and, also, the value of the temperature at which the deflection of the curve Q^{-1} (T) occurs.

To study the relationship between the anomaly of IF and the structure of bronze BK31, the investigations were made by means of dilatometric and metallographic methods.

a) The dilatometric investigations were made for cylinders with diameter 5 mm and lenght 33 mm, which were cut from the rod. The thermocouples were attached to both their ends and in the middle of the lenght. The samples were placed in the cell of the Leitz's dilatometer in the atmosphere of argon, heated to about 600°C and cooled to the ambient temperature. The relationships between the extension of the samples and their temperature have been determined. The aim of these measurements was to discover the step change of the sample lenght.

In Fig. 6a, the dilatometric plot for the steel 37AS is presented. The upper and lower curves are obtained as the results of measurements carried out during heating and cooling of the sample. The step changes of the sample length, which can be noticed on both curves, indicate the occurrence of phase transitions (ferrite-austenite). Dependence of the sample length on the temperature $l(T)$ for the BK31 bronze is presented in Fig. 6b. As before, the upper and lower curves correspond to heating and cooling of the sample, respectively. No steps similar to those in Fig. 6a can be observed on both curves. Thus, it is possible to suppose that there are no phase transitions for the bronze BK31, or that these transitions are connected with such

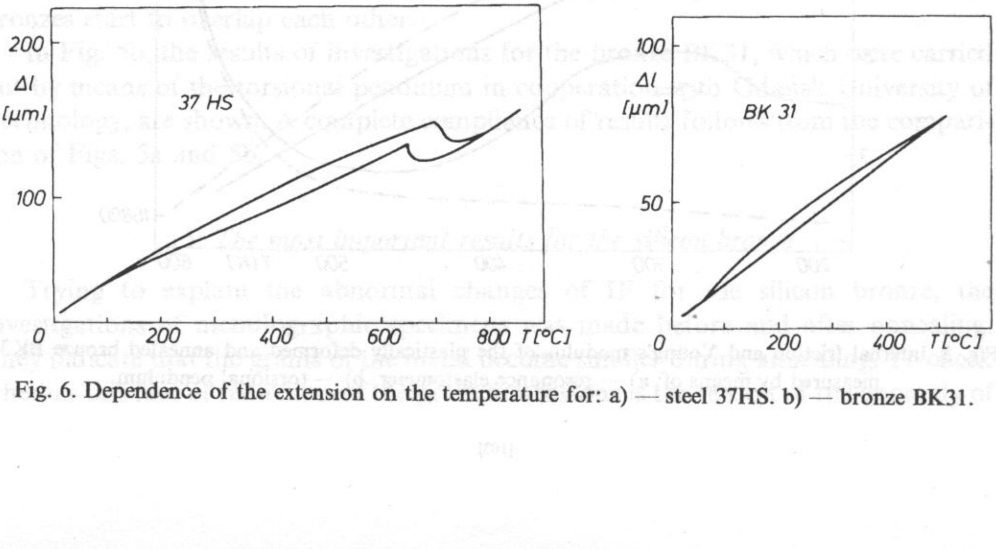

Fig. 6. Dependence of the extension on the temperature for: a) — steel 37HS. b) — bronze BK31.

a small part of the volume of the material of the sample, that the change of sample length caused by them is smaller than the sensitivity of the instrument.

b) The metallographic measurements were carried out by making the microscopic photos of the investigated sample surface to determine the grain dimensions. Two kinds of samples were applied; first — machined from the raw rod, second — previously used in dilatometric investigations i.e. annealed.

The samples were mechanically polished and then etched in the solution for developing the structure of non-ferrous metals, according to the Polish Norm PN-75/H-04512. The etched surfaces were photographed, the optical microscope at 500 times of magnification being used. Considerable differences of grain dimensions have been found in both samples. Contrary to all expectations, the grains in the annealed sample are significantly smaller than those in non-annealed one. One can think that the grains become smaller during the process of annealing. This kind of effect is no exception since, for instance during annaeling the ferrite; smaller grains of austenite appear. In the case of BK31 bronze, the origin of this phenomenon and its possible connection with the anomaly of IF have not been explained as yet.

5.1. The critical temperature

During above mentioned measurements of Q^{-1} (T), the monotonic increase of temperature was applied. However, the question appared concerning the possible explanation of the decrease of Q^{-1} (T) within the temperature range of about 100 deg (this range is bounded by two values of temperature — first value for which the Q^{-1} (T) begins to decrease, and the second one when IF attains a minimum). The first possibility considered by us was that this decrease is a gradual process, which runs successively during the temperature increase in the whole temperature range considered. The second one was connected with possible existence of the crtical temperature, attainment of which produces, in the BK31 bronze, irreversible changes reducing Q^{-1} (T) to the minimum value.

To solve this problem, investigations were carried out according to the following procedure. The sample was heated to a certain temperature and then heating was turned off, and the sample started to cool down. The measurements of IF and CE were done during the temperature increase as well as during its decrease. After complete cooling of the experimental setup (next day), the sample was heated again, but this time the temperature was a little higher than previously. At last for the third or fourth time the temperature that reduces Q^{-1} (T) to its minimum value or close to it was achieved. By subsequent approximations it has been found that the critical temperature exists, and this temperature depends on the rate of heating. So, the process of decrease of IF is activated by a certain amount of heat energy that has been supplied to the sample.

Since IF is connected with the mobility of the lattice defects, dislocations in particular, the comparative measurements of the usual samples and the ones additionally irradiated by the gamma rays dose of 40Mrad were carried out. This kind of irradiation increases the concentration of point defects, which can additionally attract the dislocations and, consequently, decrease their mobility [10, 11, 12]. After this irradiation, the depth of the decrease of Q^{-1} (T) remains unchanged, but the temperature connected with the beginning of this decrease is higher — about 70 deg — for the irradiated samples.

Critical temperatures for the non-irradiated BK31 bronze and the irradiated one are equal to 110°C (380 K) and 180°C (450 K), respectively — (see Fig. 7). Thus, this irradiation influences also the critical temperature.

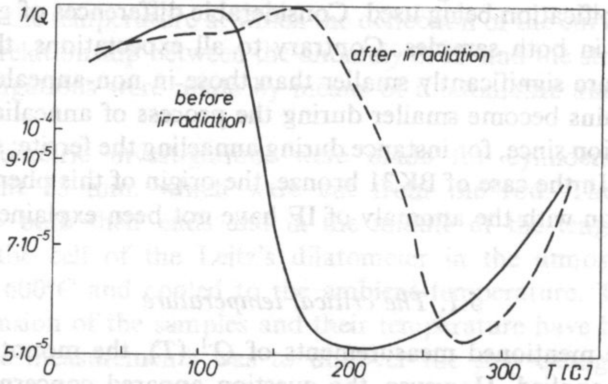

Fig. 7. Dependence of Q^{-1} (T) for irradiated and non-irradiated bronzes.

To find out how strong the connection between the anomaly of IF and the plastic deformation is, the samples with different ratios of deformation and those deformed in two steps were investigated. The first step was a plastic deformation of the raw rood in metals works, i.e. cold working. After cutting the samples off this rod and annealing them at the temperature used in the first measurement, the second step of the plastic deformation was done mechanically or by means of the hydrostatic pressure. The influence of the maximal temperature of annealing on the character of the anomaly was also examined. The samples annealed at the maximal temperature for this material, near 800 K, do not differ from those annealed at a lower temperature, even by 100 or 200 K, when the characteristics of Q^{-1} (T) for the first and second measurements are compared.

5.2. Influence of the chemical composition changes of the bronze

To investigate the importance of each component of the alloy BK31 on formation of the IF anomaly, ten laboratory-prepared alloys, with various contents of manganese and silicon, were made (Table 1). They slightly differed in compositions in comparison to the norm PN-69/H-87050, and they did not contain the additions and impurities, occuring in the raw material. The measurements of IF have not led us to the conclusion that the phenomenon of the IF decrease exists in any of these alloys in the scale comparable with BK31.

Table 1. Compositions of the laboratory-prepared alloys similar to the composition of the bronze BK31, according to the norm, [weight %].

Number of sample	Copper content [%]	Manganese content [%]	Silicon content [%]	Impurities [%]
1	95.03	1.98	2.99	—
2	95.08	1.61	2.51	—
3	96.71	1.24	2.04	—
4	97.54	0.89	1.58	—
5	98.35	0.53	1.12	—
6	96.94	1.94	1.12	—
7	96.83	1.59	1.58	—
8	96.71	1.24	2.05	—
9	96.60	0.89	2.51	—
10	96.48	0.54	2.98	—
NORM	94.0—95.3	1.0—1.5	2.7—3.5	1.0

Our analysis of the chemical composition of BK31 which was carried out with the help of the X-ray analyser, shows large differences between the reality and the norm (Table 2). Thus, the second series of the alloy, with various contents of the basic chemical elements, was prepared. This time, their compositions were based on the real one obtained from our analysis, i.e. with addition of titanium, which is not mentioned in the norm. The results of the measurements of $Q^{-1}(T)$, obtained for these alloys confirm the existence of anomalous changes of IF, which have been observed for BK31. However, these changes are smaller, because the depth of the IF decrease for laboratory-prepared alloys is lower than for the raw material, i.e. originating from

Table 2. Compositions of the laboratory-prepared alloys similar to the real composition of the bronze BK31 [weight %]

Number of sample	Copper content [%]	Manganese content [%]	Silicon content [%]	Titanium content [%]	Impurities [%]
1	97.12	1.35	1.53	—	—
2	98.27	0.20	1.53	—	—
3	97.93	0.50	1.53	—	—
4	98.45	1.35	0.20	—	—
5	98.15	1.35	0.50	—	—
6	100.00	—	—	—	—
7	96.47	1.35	1.53	0.65	—
BK31	94.7	1.30	1.50	0.60	1.90

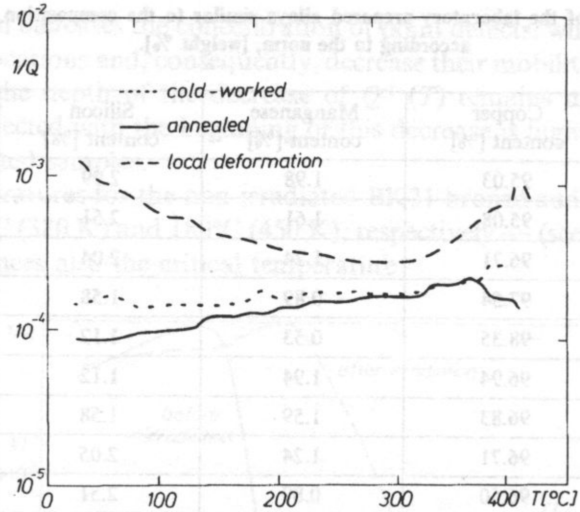

Fig. 8. Dependence of Q^{-1} (T) for the sample No. 10 from Table 1. This sample was deformed to a different extent.

metalurgical works (Fig. 8). The ratio of plastic deformation is undoubtedly higher for raw rods as a result of cold working in metal works than for the samples machined from cast billet and squeezed in the laboratory. Thus, the increase of the ratio of deformation intensifies the anomaly of Q^{-1} (T).

It is possible to assume that the interactions between the basic components of the alloy, eventually in the presence of the vacancies, are the origin of the described anomaly of IF. In the metal, the concentration of vacancies significantly increases when the deformation and temperature increase, and decreases after annealing. The various phases of such compounds as for instance Cu-Si, Cu-Ti, Mn-Ti, Mn-Si and others, can be these components. Our investigations will be continued to confirm the above suggestions.

6. Conclusions

1. The measurements of IF and CE for several metals, which were carried out at the beginning of the measurements by means of the resonance elastometer, gave us the results complying with the references. This confirm the opinion about proper functioning of the measurement equipment and reliability of the measurements made. These measurements enabled us to find the important differences in the value of IF occuring in the silicon bronze and in other materials tested.

2. There is an interesting property of the silicon bronze; the approx. tenfold decrease of IF occurs in the plastically deformed state of this bronze at the temperature in the range of 400... 500K. This phenomenon does not appear in the annealed samples; then Q^{-1} (T) increases monotonically.

3. The decrease of IF at 400... 500K should be caused by the decrease of the dislocation motion, i.e. the appearance of additional obstacles which block this motion. This fact can be connected with attraction of the dislocations by the point defects or complexes formed by the atoms of silicon, manganese and, eventually, titanium and by vacancies.

4. The obstacles of small dimensions even those which are comparable with the dimensions of a few atoms are sufficient to impede the dislocation motion. On the other hand, the possibility or direct recognition of such small obstacles is very small in the highly complicated structure as BK31, even with the help of the electron microscopy. Therefore, indirect methods, based on investigations of macroscopic properties of the material, are necessary.

5. There is a critical temperature for plastically deformed BK31, which depends on the time of annealing. When this temperature is attained the process of significant decrease of IF can not be stopped solely by the temperature decrease.

6. Comparison of the temperature characteristics of the anomaly of IF for the irradiated and non-irradiated bronzes indicates that the starting point of this anomaly is shifted by about 80 deg towards higher temperature in the case of application of the irradiation.

7. The described feature of the temperature characteristic of IF for the silicon bronze is a unique phenomenon. The reasons of this phenomenon are not known as yet. We intend to carry on further investigations to explain that phenomenon.

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