

## LOW-TEMPERATURE CREEP OF LITHIUM IN THE POLYMORPHOUS TRANSFORMATION RANGE\*

I. A. GINDIN, V. M. GODZHAYEV, M. B. LAZAREVA and YA. D. STARODUBOV

Physico-technical Institute, Academy of Sciences, U.S.S.R.

(Received 9 March 1965)

Creep of lithium has been studied at temperatures of 300, 180 and 77°K, which embraced the polymorphous transformation range, and the electrical resistivity of the specimens has been measured in the process of creep. It was found that at 77°K the logarithmic law of the transitory stage of creep is satisfied right up to those stress values at which there is either no polymorphous transformation, or only a slight one. Even for slight stresses, the creep curves of single-phase specimens at 300°K do not follow the logarithmic law. During creep at 77°K it has been found that there is a resistivity maximum for the original specimens, with a monotonic drop on those which have undergone prior deformation at 77°K.

A number of works have dealt with low-temperature creep [1-5]. These works cover creep in mono- and polycrystalline metal specimens with a stable crystalline structure right up to helium temperature. On the basis of experiments Mott in [6-8] has put forward a dislocation theory of low-temperature creep and has given a logarithmic substantiation of the time dependence of deformation.

In the present work it was found that alkaline metals such as lithium and sodium undergo a polymorphous transition with martensitic kinetics under conditions of low-temperature static deformation [9-12]. On extension in liquid nitrogen, for instance, the b.c.c. lattice of lithium is partially transformed to an f.c.c. one. Typically, in the process of deformation the specimen goes over from the single-phase to a "two-phase" state with a continuously altering quantitative phase ratio. It is therefore interesting to study the creep of lithium in the temperature range of this polymorphous b.c.c.-to-f.c.c. transformation on deformation. The present work was an investigation of the nature of the creep of lithium at 300, 180 and 77°K, temperatures which cover the polymorphous transformation range.

## MATERIALS, SPECIMENS AND PROCEDURE

The object of the investigation was polycrystalline lithium 99.3 per cent pure. Specimens for the creep tests were made by extrusion through an aperture 3 mm dia. at room temperature. Spherical heads like the inserts of chucks [13] were riveted on the ends of the cylindrical specimens (the test length of which was 100mm).

To avoid oxidation all the operations of preparation were carried out in kerosene. To remove the resultant cold work we used a long anneal in kerosene at room temperature. The oxide film was removed by etching in methyl alcohol.

The low-temperature creep was brought about at a constant load on the apparatus described in [13]. The deformation was measured by means of an optical pick-up with a photographic recording of the "elongation-time" graph. The creep tests were performed at 300, 180 and 77°K. The creep curve was determined as

\* *Fiz. metal. metalloved.*, 21, 4, 600-607, 1966.

dependent on the magnitude of the initial stress  $\sigma_0$ . The stress level\* was varied from 0.1-1.8 kg/mm<sup>2</sup>. Creep curves were taken and the instantaneous deformation sector and transition area studied. The test lasted from 5-50 hr.

The specimens were creep tested in the following structural stages; (1) with a stable b.c.c. lattice, at 180 and 300°K; (2) with supercooled b.c.c. lattice at 77°K; (3) after prior deformation of 35 per cent in liquid nitrogen, perpendicular to the tension axis at 77°K.

At the same time as the low-temperature creep the electrical resistivity was also measured in specimens in the single-phase and two-phase initial states. Spring clamps were used for the electrical contact. For these measurements the grips were insulated from the apparatus with fluorplast. The behaviour of the microstructure was also studied during the low-temperature creep.

## RESULTS AND DISCUSSION

1. *Creep of original polycrystalline lithium.* The mechanical creep curves for lithium specimens at 300, 180 and 77°K have an instantaneous deformation section, a transitory stage of creep and a stage of steady flow. As an example Fig. 1 shows the creep curves of Li specimens at 77°K.

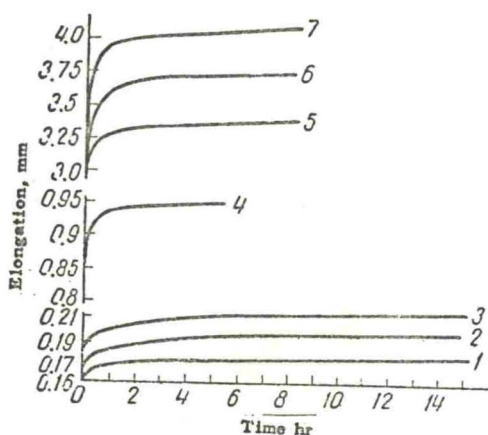


FIG. 1. Mechanical creep curves of lithium specimens at 77°K and different initial stresses  $\sigma_0$ :

1—0.1; 2—0.2; 3—0.3; 4—0.6; 5—1.2; 6—1.3 and 7—1.5 kg/mm<sup>2</sup>

As the testing temperature falls the magnitude of the instantaneous deformation, transitory stage and rate of steady-state creep diminish for any given stress value. For instance, at  $\sigma_0 = 0.1$  kg/mm<sup>2</sup> the instantaneous deformation was 640 and 170  $\mu$  at 300 and 77°K respectively; the size of the transitory flow stage was 310 and 16  $\mu$ ; the rates of steady-state creep differed 45 times. An increase in stress has the same effect on creep as a rise in temperature. But the stress has the greater influence. Some of the creep parameters (stress dependence of the steady-state flow rate for instance, Fig. 2) have non-monotonic dependence. Like the steady flow rate, the magnitude of the instantaneous deformation and deformation at the transitory stage undergo a sudden change at stresses above 0.6 kg/mm<sup>2</sup>. This may be due to the polymorphous transition at the initial stage of creep which takes place as from a given stress level. As a result of the partial transition and formation of complex system, the rate of steady creep practically ceases to

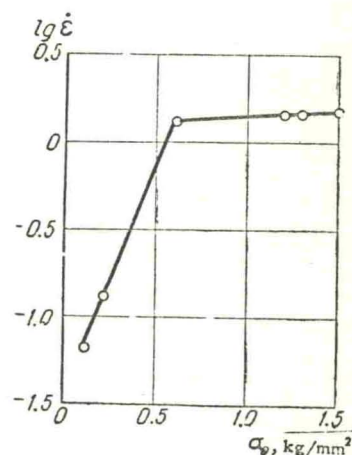


FIG. 2. Rate of steady-state creep for lithium specimens at 77°K as a function of the initial stress.

\* Under static tests in this temperature range the yield point was 0.2-0.6 kg/mm<sup>2</sup>.

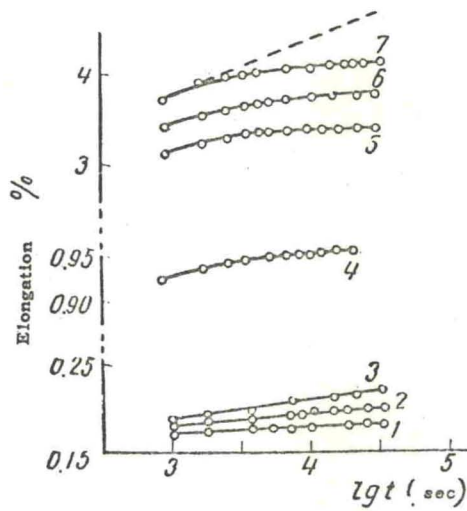


FIG. 3. Creep curves for lithium at 77°K, various  $\sigma_0$ :  
 1—0.1; 2—0.2; 3—0.3; 4—0.6; 5—1.2; 6—1.3; 7—1.5 kg/mm<sup>2</sup>

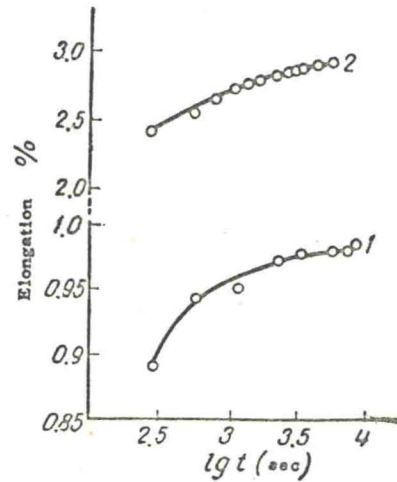


FIG. 4. Creep curves of lithium at various temps and stresses.  
 1— $\sigma_0 = 0.1 \text{ kg/mm}^2$ ,  $T = 300^\circ \text{K}$ ; 2— $\sigma_0 = 1.0 \text{ kg/mm}^2$ ,  $T = 180^\circ \text{K}$ .

depend on the stress magnitude above  $\sigma_0$ .

Fig. 3 shows the results of the creep of lithium at 77°K in semilogarithmic coordinates: relative deformation-log time at different stress levels. It can be seen that low-temperature creep definitely follows the logarithmic law at stresses right up to 0.3 kg/mm<sup>2</sup>. At 0.6 kg/mm<sup>2</sup> and above the logarithmic dependence is not satisfied.

As expected, at 180°, and more particularly at 300°K (i.e. at quite high temperatures) despite the stable crystalline structure of the metal, creep in lithium does not follow the logarithmic law (Fig. 4). But comparing Figs. 3 and 4 it can be seen that as the temperature falls the deviation from the logarithmic law diminishes, even at quite considerable stresses.

The kind of interdependence observed between the parameters and creep curves in the case of annealed lithium can be explained as follows. The low-temperature creep of lithium at stresses below the yield point (0.5-0.6 kg/mm<sup>2</sup>) occurs at the stages of instantaneous deformation and transitory yield by movement of dislocations, their exhaustion in the original lithium lattice and accumulation at various different barriers. In this case the time dependence of creep is very well described by the logarithmic law ( $\epsilon = a \lg t + c$ ). If the stress is increased to levels above the yield point at 77°K creep will occur, accompanied by the polymorphous transformation from the b.c.c. to h.c.p. lattice\*. At this stress level there is also a big increase in the instantaneous deformation and transitory yield.

As follows from the electrical resistivity data (see Fig. 7) the rise in instantaneous deformation during creep cannot be due to an increase in stress alone, but mainly to an increase in the mobility of the lattice as a result of the strain transition which is taking place. But the reduction in the deformation at the transitory stage as compared with that described by the logarithmic law must be ascribed to a drop in the mobility of dislocations due to the partial polymorphous transition. Thus, while the deviation from the logarithmic law for the transitory stage of creep in specimens tested at 300 and 180°K is due to the relatively high testing

\* To a certain extent the transition from b.c.c. to h.c.p. is also possible, since this follows from measurements of damping capacity at low temperatures [12].

temperature, even at low stresses, in the case of creep in liquid nitrogen and stress of 0.6 kg/mm<sup>2</sup> and above, the departure from this regularity seems to be due to the formation of a single-phase structure as a result of the polymorphous transition.

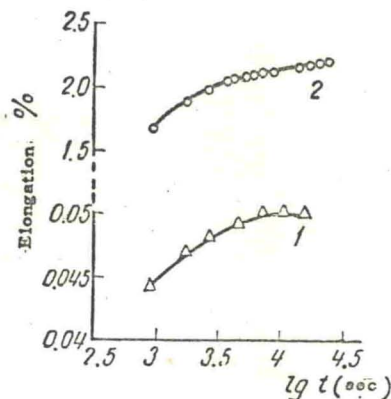


FIG. 5. Creep curves of lithium at 77°K after prior 35 per cent deformation in liquid N<sub>2</sub>.

1 -  $\sigma_0 = 0.3$       2 -  $\sigma_0 = 1.5$

2. *Creep of lithium specimens after prior deformation at 77°K.* Fig. 5 shows the creep curves of lithium specimens at 77°K after prior deformation by 35 per cent in liquid nitrogen.

The prior deformation causes a partial b.c.c.-to-f.c.c. transition, and the movement of dislocations during creep takes place from the very beginning in a complex two-phase system. This explains the low value of deformation at the initial stage of creep as compared with the same section on the original specimen tested in creep at the same level of initial stress, 1.5 kg/mm<sup>2</sup>.

Like the specimens of lithium with a complex multi-phase structure formed in the process of low-temperature creep under stresses about the yield point, specimens which have undergone prior deformation at 77°K and have a b.c.c. + f.c.c. structure, exhibit creep which does not follow the logarithmic law. In both cases the prior strengthening treatment causes a more sudden drop in creep at the unsteady stage.

One can imagine the creep curve  $\epsilon(\lg t)$ , as consisting of separate parts parallel to separate beams  $\epsilon(\lg t)$ , obtained at low stresses. Then the degree of strengthening due to the two-phase state of the system can be assessed from the fact that the final section of the curve at 0.6 kg/mm<sup>2</sup> has the same angle of slope as the beam obtained at  $\sigma_0 = 0.1$  kg/mm<sup>2</sup> (Fig. 3).

Studies of creep under low stresses, on specimens which have undergone prior deformation, suggest that the infringement of the usual form of dependence  $\epsilon(\lg t)$  is not connected with the magnitude of the stress, but is due to the complexity of the phase composition in the system studied.

3. *Steady-state stage of low-temperature creep.* In earlier investigations [1, 3] the creep tests were usually limited to certain periods of time (of the order of several tens of minutes) embracing the instantaneous jump in deformation and transitory stage of creep. Sometimes the creep curves (at low  $\sigma_0$ ) had sections where there was a withdrawal to the horizontal branch ( $\dot{\epsilon} = 0$ ).

In contrast to those works, our studies of creep in lithium at low temperatures were carried out in a wide range of stresses and testing times (up to 50 hr). At low stresses the creep curves at 77°K (see Fig. 1) show a transition from the unsteady state of flow to practically complete decay of the creep. On the curves

taken at stresses of 0.6 kg/mm<sup>2</sup> or above there are distinct sections of steady-state creep, for which the flow rates are very high. This shows that, even at low temperatures, recovery processes take place in lithium under conditions of slow plastic deformation. The most probable mechanism of recovery in low-temperature creep is the activationless process of the detachment of dislocation pile-ups, such as grain-block boundaries and interfaces, from obstacles. Thus the steady-state stage of creep can be represented as a successive alternation of horizontal and vertical sections.

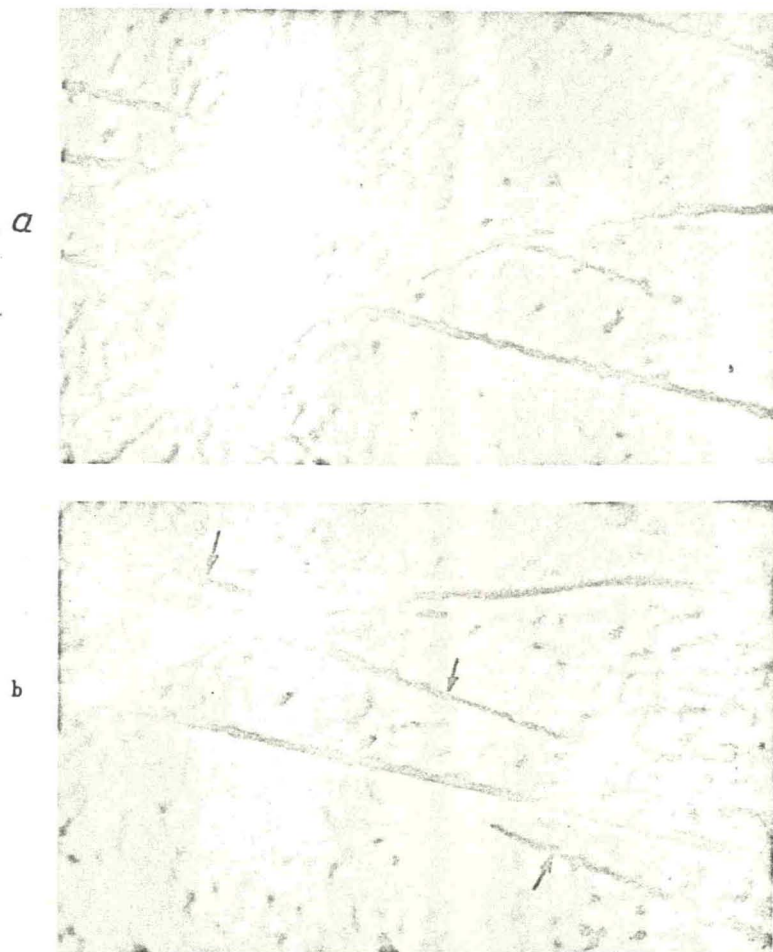


FIG. 6. Development of slip bands at the steady stage of creep at 77°K: a - micropattern after  $\sigma_0 = 0.65$  kg/mm<sup>2</sup>; b - micropattern after 7 hr creep ( $\sigma_0 = 0.65$  kg/mm<sup>2</sup>): X 750.

Low-temperature deformation is also known to be connected with the formation of a large number of point defects, i.e. vacancies which, as we know from experiments in the recovery of electrical resistivity of cold-deformed metals [14], have very high mobility. The redistribution of the point defects, which amounts to the formation of paired vacancies, vacancy pile-ups, and their migration to dislocations, may also lead to recovery during the low-temperature creep of lithium.

Finally, some contribution to the process of recovery is made by stress relaxation by the additional development of existing inclusions of the low-temperature phase. As the metallographic studies show, at 77°K, in creep, bands arise in which the f.c.c. phase is concentrated, and these develop rapidly as an after-effect of stress relaxation at the apex of the wedge-shaped band. This is quite obvious if one compares Figs. 6a and 6b, where the elongations of martensite bands (indicated by arrows) occurred after 6 hr creep at 0.65 kg/mm<sup>2</sup> in the steady stage.

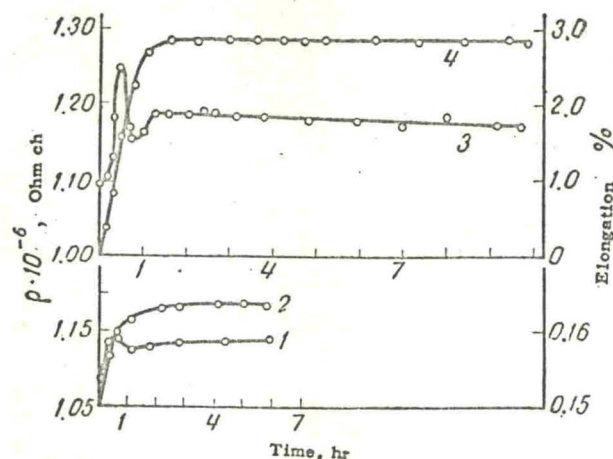


FIG. 7. Creep curves of lithium specimens at 77°K (2 and 4) and variation in electrical resistivity during creep (1 and 3) under different stresses:

$$1 \text{ and } 2 - \sigma_0 = 0.2 \text{ kg/mm}^2 \quad 3 \text{ and } 4 - \sigma_0 = 1.2 \text{ kg/mm}^2$$

4. *Resistivity of lithium specimens.* At the same time as the creep curves were taken at 77°K the resistivity of the original specimens was measured, and of those which had been deformed at liquid nitrogen temperature.

There is a big difference in the time dependence of the resistivity of these two batches. For the original specimens, which had undergone the low-temperature transition in the process of creep, the resistivity showed a time variation as follows (Fig. 7). The beginning jump and the beginning of the transition stage of creep corresponded to an increase in the volume resistivity and the point where a maximum was reached followed by a drop and rise up to a certain level, which remains unchanged at the transitory and steady-state stages of creep. The height and position of this jump in relation to the different stages on the creep curve depend on the initial stress. As the stress rises so does the maximum, and then gradually shifts towards the initial stage of unsteady creep; there is a very distinct second rise in resistivity close to the transition point to the steady-state stage of flow.

In all cases, after transition to the steady state of creep the resistivity was higher than in the initial state. But the size of the increment also depends on the creep stress and increases parallel to it. Typical creep curves ( $\sigma_0 = 1.5$  and  $1.8 \text{ kg/mm}^2$ ) and resistivity curves are shown in Fig. 8 for specimens after prior

deformation by 35 per cent at 77°K.

The time dependence of the resistivity of pre-deformed specimens shows a continuous drop in  $\rho$ . The maximum rate of the drop is found in the initial section of the unsteady stage. At this stage the size of the drop is 4 per cent. It is interesting that there is a continuous drop in  $\rho$  even in the process of creep at high stress in the accelerated stage just prior to failure.

The difference in the form of the dependence of resistivity on the pre-history of the specimen is the result of the difference in the initial structure. The rise in the resistivity of originally annealed specimens is due both to the process of plastic deformation (mainly at low stresses), and to the polymorphous b.c.c.-f.c.c. transition which takes place in the initial stage of flow, with martensitic kinetics [12]. The role of the latter in increasing  $\rho$  increases with the creep stress.

The different course of the resistivity in the two batches of specimens is due to the fact that the pre-deformed specimen is already strengthened, and the original one is being strengthened in the process of creep. Since the phenomenon of recovery may be observed as a result of the strengthening which in the one case takes place before creep and in the other during it, it will begin at different stages of creep in the two different batches. In the original specimens therefore, the processes of strengthening and recovery should be parallel and lead to the formation of a maximum on the curve for the time dependence of resistivity.

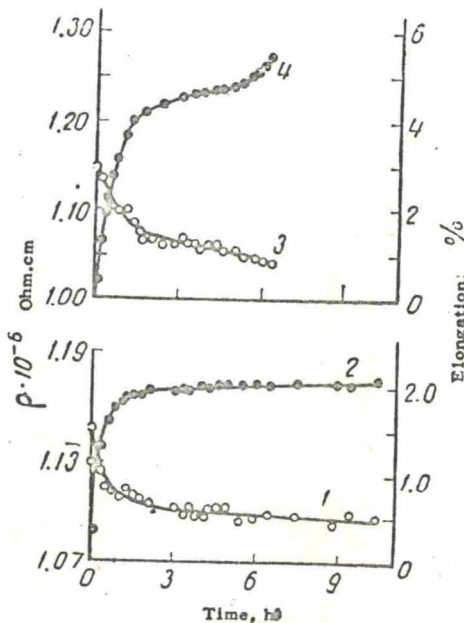


FIG. 8. Creep curves (2 and 4) and variation in electrical resistivity (1 and 3) of lithium in the process of creep at 77°K, for specimens predeformed to 35 per cent at 77°K:

$$1 \text{ and } 2 - \sigma_0 = 1.5 \text{ kg/mm}^2 \quad 3 \text{ and } 4 - \sigma_0 = 1.8 \text{ kg/mm}^2$$

The shift of the  $\rho$  peak to the left as  $\sigma_0$  rises (Fig. 7) is due to the fact that recovery begins early and is more vigorous at higher stresses. Due to the work hardening in specimens which have been deformed at 77°K, recovery begins at the very beginnings of the process of creep and continues right up to the steady stage of flow.

## CONCLUSIONS

1. In prolonged low-temperature creep of lithium the curves have the three stages characteristic of normal creep, i.e., instantaneous deformation, transitory stage and steady stage of flow.

2. At room temperature\* and low stresses ( $0.1 \text{ kg/mm}^2$ ) the creep curves of single-phase specimens do not follow a logarithmic law. At low temperatures ( $77^\circ\text{K}$ ), in lithium as in a number of other metals [2], the logarithmic law of creep holds right up to the stress levels at which the polymorphous transition is either totally absent or very weak. Beginning from a certain stress ( $0.5\text{-}0.6 \text{ kg/mm}^2$ ), sufficient for the b.c.c.-to-f.c.c. transition, the logarithmic law of the transitory stage of creep is not satisfied. The same thing is found in specimens after prior deformation at  $77^\circ\text{K}$ , even under conditions of creep with low stresses ( $0.3 \text{ kg/mm}^2$ ).

3. The main b.c.c.-to-f.c.c. transition takes place in the period of instantaneous deformation in creep and follows martensitic kinetics.

4. The low-temperature creep curves of lithium specimens have a steady stage of creep. Therefore, even at liquid nitrogen temperature, recovery as well as strengthening processes occur in lithium specimens under slow deformation. These processes seem to be accelerated by the development of martensitic bands at the steady-state creep stage.

5. By measuring the resistivity at  $77^\circ\text{K}$  we have found a maximum for the initial specimens and a monotonic drop in resistivity for specimens predeformed at  $77^\circ\text{K}$ . This difference in the resistivity is due to the different ratios of the processes of strengthening and recovery for the two different batches of specimens.

## REFERENCES

1. W. Meissner, M. Polanyi and E. Schmid, *Z. Phys.*, 66, 477 (1930).
2. O. H. Wyatt, *Proc. Phys. Soc.*, B66, 459 (1953).
3. J. M. Glen, *Phil. Mag.*, 1, 400 (1956).
4. P. R. Thornton and P. B. Hirsch, *Phil. Mag.*, 3, 738 (1958).
5. N. S. Stewe, *J. Appl. Phys.*, 3, 30, 450 (1959).
6. N. F. Mott, *Proc. Phys. Soc.*, B64, 729 (1951).
7. N. F. Mott, *Phil. Mag.*, 44, 742 (1953).
8. N. F. Mott, *Phil. Mag.*, 1, 568 (1956).
9. C. S. Barrett, *Phys. Rev.*, 72, 194, 245 (1947).
10. I. A. Gindin, V. G. Lazarev and Ya. D. Starodubov, *Fiz. metal. metalloved.*, 10, 472 (1960).
11. I. A. Gindin, V. G. Lazarev and I. R. Khredchuk, *Fiz. metal. metalloved.*, 16, 793, 795 (1963).
12. R. I. Garber, I. A. Gindin and L. A. Chirkina, *Fiz. metal. metalloved.*, 20, 603 (1965).

---

\* For lithium  $300^\circ\text{K}$  is a high temperature.



13. I. A. Gindin, S. F. Kravchenko, Ya. D. Starodubov and V. M. Godzhayev, *PTE*, 3, 169 (1963).
14. *Sb. Polzuchest' i vozvrat.* (Collection. *Creep and recovery*) For. Lit. Pub. Hse, Moscow, 21, 127 (1961).

characteristic of

base specimens  
er metals [2], the  
sition is either  
for the b.c.c.-to-  
same thing is  
w stresses

formation in creep

creep. Therefore,  
lithium specimens  
artensitic bands

specimens and a  
resistivity is due to  
edges of specimens