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Low-Temperature Cycling and its Effect on Structural State and Mechanical Properties of Metals and Alloys

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A brief review of the experimental data available on low-temperature cycling effect on structure and cryogenic mechanical properties of alloys and metals of different lattice type (fcc, bcc, hcp) is given.

The papers where the direct quantitative investigations (electronmicroscopy, x-ray, metallography) of structural state changes at cryogenic temperatures and their influence on mechanical properties of metals and alloys have been studied were considered most carefully. The efficiency of low-temperature cycling (LTC) for improving operating characteristics of materials is shown on the example of different structural metals and alloys (titanium, beryllium, stainless steels, etc.).

Shrnujeme souborným způsobem výsledky získané studiem termomechanického cyklování na materiálech a slitinách s různou mřížkou (KPLC, KPRC, HTU). Hledá se fyzikální mechanismus, odpovědný za změny fyzikálních vlastností za velmi nízkých teplot. Jsou vytvořeny modely relaxace termických napětí skluzem a dvojčatěním.

Изучается влияние термомеханического циклирования на механические свойства металлов и сплавов с различной решёткой (ГЦК, ОЦК, ГПУ). Ищется микрофизический механизм действующий во время циклирования. Проводится тщательный анализ именений в структурном состоянии и свойствах в процессе термического циклирования (наружных и внутрених). Термомеханические напряжения, возникающие при температурном циклировании при долее высоких температурах аккомодируются диффузионными процессами. При низких температурах этот процесс не имеет места. Для низких температур предлагается новая модель релаксации термических напряжений скольжением и двойникованием.

Introduction

The structural elements and assemblies of cryogenic engineering suffer repeated influences from LTC and loads during operation. The structural state of metals and alloys changes under thermomechanical influences and leads to subsequent variations

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in the totality of structure-sensitive physical properties and especially in the mechanical properties of these materials. Thus investigation of the regularities of structural state evolution during cycling and revealing the physical mechanisms responsible for those changes are the necessary preconditions for the solution of the problem of material manufacturing with the present properties stable to different thermomechanical effects and able to work at cryogenic temperatures for a long time.

It should be mentioned, that nature of the thermomechanical influence on structure and mechanical properties of metals and alloys is studied insufficiently and is explained by the variety of facts determining this influence. The most work concerning thermocycling effect on material structure and properties was carried out in the temperature range above 300 K. Below 300 K such investigations were considerably fewer and mainly of qualitative character.

It should be emphasized, that although the physical causes of thermal stresses in the range of high and low temperatures of cycling are the same, there is principally an important difference in relaxation mechanisms of these stresses in the above mentioned temperature ranges.

During material cycling at high temperatures (HTC) an essential role in stress decrease play diffusion and other thermally activated processes which are considerably supressed by low-temperature cycling. During LTC the thermal stress relaxation occurs mainly due to plastic deformation processes (slip, twinning) and phase transformations.

Thus, the LTC effect on material structure and properties has a series of specific pecularities, and regularities true for HTC cannot be fully extended on the low-temperature region.

This, a rather brief review, consideres the factors influencing the LTC efficiency and analysis of the most important experimental results concerning the cycling effect on structure and mechanical properties of structural materials and alloys of different lattice types (bcc, fcc, hpc).

Factors influencing the LTC efficiency

The cause of thermal stresses appearing during temperature variation are the obstacles to thermal expansion (contraction) of materials [1].

The highest thermal stresses occur in the case, when material undergoes temperature gradient (thermal shock). These conditions occur when material with uniform temperature is immersed into medium of a different temperature [2]. Here stresses of the 1st kind may appear balancing within the given body.

Different grain orientations prevent the free change in a separate grain size in polycrystals during temperature variations. Here stresses of the 2nd kind occur balancing within the grain sizes. Up to elastic limit thermal stresses increase in proportion to the increase in constant deformation.

For the anisotropic materials level of the 2nd kind stress is determined by the

difference between the maximum and minimum cycle temperature and by the aniso-tropy degree of thermal expansion coefficients.

Change in the material structural states and properties during LTC depends on many factors which can be divided into external and internal ones.

The external factors:

(a) shape of the sample,

(b) cycling parameters (temperature gradient, heating and cooling rate, time of keeping at the given temperature),

(c) thermophysical properties of cooling medium,

(d) presence of the external load or its absence.

The internal factors:

(a) crystal lattice type of metal or alloy,

(b) initial structural state (degree of structural perfection),

(c) thermophysical properties of material,

(d) material tendency to phase transformations,

(e) stacking fault energy of dislocations,

(f) anisotropy degree of material physical properties.

(g) acting types of plastic deformation (slip, twinning).

Depending on the above external and internal factors the thermal stress relaxations occuring on metals and alloys during LTC can proceed differently and this stimulates a various character of structural state formation, and consequently of structure-sensitive physical properties. Consider the LTC methods and mechanisms of thermal stress decrease.

Methods of cryogenic thermocycling

Consider briefly the LTC peculiarities. More details on LTC techniques one can find in references given below.

Cycling effects on structure, mechanical properties, shape change, fatigue of materials have been studied by various techniques. However these techniques were mainly developed for the high temperature cycling. The cycling in low-temperature range has a number of peculiarities associated with that the sample cooling is usually acomplished by their immersion into cryogenic liquid. This creates sharp temperature gradients which lead to large internal stresses, especially in the case of massive samples. Together with the sample a part of loading device is inevitably cooled and this complicates measuring of stress and deformation. During reheating the moisture can condensate on sample and loading system. Therefore reheating should be carried out in the anhydrous gaseous medium of controlled composition.

Now a relatively small number of designs are known which allow LTC of materials in the range of low and superlow temperatures. The most perfect design for the cryogenic LTC was made at FTINT Ukr. SSR Acad. of Sciences[3−6]. This device allows thermocycling up to 4.2 K with the programmed sample loading and cooling.

Relaxation on thermal stresses by plastic deformation

The thermal stresses due to LTC can decrease by plastic deformation. In metals and alloys where slip is the characteristic type of plastic deformation the thermal stress relaxation occurs by migration of perfect dislocations. In twinning crystals the thermal stress relaxation proceeds by the nucleation and motion of twinning dislocations.

Relaxation by slip

Boas and Honycomb[7-10] were the first who studied LTC plastic deformation in crystalline materials. The showed that during cycling thermal stresses lead to slip lines appearing in tin, cadmium, zinc polycrystals and their alloys. The slip lines appear in separate grains already after one or two cycles. With the increase of cycle number the slip involves more and more grains and the slip lines available widened.

Similar results are now known for a great number of metals and alloys both of anisotropic and isotropic lattices[11-26]. The data obtained are in good agreement with dislocation ideas of plastic flow taking place in crystalline grains only under external stresses[27-30]. This flow is especially active during LTC. This is explained by that the thermal stresses, appearing during cycling in the low-temperature range, cannot decrease through diffusion and recrystallization, as they do at high temperatures, and relax only on account of plastic deformation[31]. The experience shows that this deformation at $T < 0.5_{melt}$ (T_{melt} is the temperature of metal melting) is mainly of dislocation character. Relaxation of the thermal stresses, appearing locally in poly-and single crystals during low-temperature cycling, depends on dislocation multiplication in the regions of stress concentrations and their motion under total internal stresses.

During cycling to lower temperatures (to 77 K) the fine slip lines prevail in grains[24] and the slip is multiplet and inhomogeneous. The multiple slip involves also the slip systems, which do not take part under usual conditions[24,32]. So, while cycling the cadmium in the range 283 ⇄ 458 K slip in grains occurs only in the $\langle 11\bar{2}0 \rangle (0001)$ system and during LTC in the range 281 ⇄ 87 K an active slip is also observed in grains of non-basic slip systems[24]. Double slip begins in many grains already after first cycles. With cycle number increase the slip begins on three and more slip planes in one grain. The deformation becomes extremely inhomogeneous: regions where different slip systems act are observed within one grain. As a rule the slip line density is maximum near grain boundaries. With the increase in cycle number one slip system develops most intensive in grains of multiplet slip and supresses the other[24].

The intensive multiplet slip after first cycles and inhomogeneous deformation in grains were also observed in zinc treated in the temperature range 293 ⇄ 88 K and in tin during cycling in the range 300 ⇄ 77 K. It should be mentioned that deforma-

tion within the grains during cycling is similar to that in grains of polycrystals under external load[33,34]. This is especially mentioned in[13], where LTC effect (283 ⇄ 77 K) was studied in polycrystalline tin. It is indicated that deformation of the multiplet character and strongly dependent on grain orientation takes place in tin near grain boundaries during LTC as well as in process of usual deformation. As a result the grains are deformed differently: besides the strongly deformed grains the grains appear which have no slip traces. When studying the LTC effect on alloy structures, containing phases of different thermal expansion coefficients, Khenkin et al.[18] found that thermoelastic stresses, appearing at cooling during cycling in the range 293 ⇄ 77 K, interact with the internal stress field and lead to a considerable plastic deformation of alloy.

The LTC is also characterized by the grain grinding that being direct consequence of multiple slip within grains. The new grain formations occur usually in places of high stress concentrations (grain joint, change in the boundary etc.). During LTC of cadmium the subgrains in some places (more often at grain boundaries or joints) appeared already after a few cycles. With the cycle number increase the grinding process enhances, number of grains, involved in this process, grows. After 50 cycles the most grains are broken into subgrains. The grain grinding in zinc polycrystals during cycling in the range 293 ⇄ 77 K was observed in[17,21]. An active subgrain formation is observed during cycling tin[13,23], lead[13] aluminium[14] and a number of structural materials[25−27]. In all cases grinding occurs mostly at grain-boundary angles between blocks increased up to formation of the large angle grain boundaries and within the initial grain some new grains appeared[24].

It should be mentioned, that in the case of LTC of single crystals there is a considerable difference compared to LTC of polycrystals. So, it was shown[35] that cycling in the range 300 ⇄ 77 K does not practically influence the initial structural state of zinc single crystals, while in polycrystalline zinc an intensive grain grinding occurs in subgrain, as mentioned above[17,21]. This difference in LTC effect seems to be due to that the single crystals do not have interfaces (of grains) on which the stresses appear because of different expansion coefficients, characteristic of polycrystals. This conclusion is confirmed by the fact that the processes of slip and change in structural state during LTC in polycrystals begin mainly at grain boundaries and their joints[8,24].

Values of the stresses (σ_T − arising during cryogenic cycling because of different thermal expansion coefficients ($\Delta\alpha$) along various crystallographic directions we may estimate using the formula:[36]

$$\sigma_T = E \,.\, \Delta\alpha \,.\, \Delta T,\tag{1}$$

where E is the Young's modulus, $\Delta T = T_2 − T_1$ − difference between cycling temperatures. The estimated σ_T values for some fcc metals and alloys where the anisotropy during LTC in the range 77 ⇆ 300 K is essential are given in Table 1.

The σ_T values for Zn and Cd exceed $\sigma_{0,2}$ and for Be and Ti + 5%Al are by the

Table 1

Material	σ_T (MPa)	E (MPa $\times 10^3$)	$\Delta\alpha = \alpha_{h,K,l} - \alpha_{h_2,K_2,l_2,K^{-1}}$	$\Delta T = T_2 - T_1\ K$
Zn	~800	80	47×10^{-6}	2.23×10^2
Cd	~400	50	32×10^{-6}	2.23×10^2
Be	150	320	2.2×10^{-6}	2.23×10^2
Ti + 5%Al	70	135	2.3×10^{-6}	2.23×10^2

Table 2

T (K)	N	$\sigma_{0,2} \cdot 10^7$ (Pa)	$\sigma_\beta \cdot 10^7$ (Pa)	δ (%)
300	0	54	61	5
77	0	79	103	7
4.2	0	87	112	7
4.2	300	100	123	9

order of magnitude commensurable with the critical shear stress values, at which the macroscopic plastic deformation begins and results in the change of material structural state during LTC.

In should be mentioned that presence of internal stresses from different concentrators (inclusions, dislocation pile-ups, grain boundaries etc.) increases the value of stresses acting in local places compared to those estimated in Table 1.

Relaxation of the thermal stresses through perfect dislocation migration manifests itself variously. So, for example, in Ti + 5%Al structural alloy decrease of thermal stresses arising during LTC can occur by formation of helical dislocations[36]. Fig. 1 shows that the helical dislocation begins at the grain boundary $\Gamma - \Gamma$ and distributes in the $[11\bar{2}0]$ direction. The stage preceding the formation of helical dislocation is shown in Fig. 1(b), where a number of dislocation loops in $[11\bar{2}0]$ is seen, whose screw components at twisting form helicoids. Knowing geometrical parameters of helicoids (length L, pitch λ and cylinder radius r) one may estimate the values of total stresses involving the helicoid formations[37].

Calculation shows, that $\tau = G/1000 = 50$ MPa, what is essentially lower than the theoretical elastic limit $G/10$ and is consistent with the thermal stress value of about 70 MPa (see Table 1) obtained with (1). A good coincidence of the estimated stress values shows that the helical dislocation formation is a mechanism strictly controlling relaxation of thermomechanical stresses occuring at grain boundaries during LTC.

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Mechanical twinning during LTC takes place at the same time as the transgranular slip and grinding[13,16,19,21,23]. The most intensive twinning occurs in metals with a low number of slip systems[16,38]. An intensive twinning was observed in cadmium polycrystals during 77 $\leftrightarrows$ 300 K cycling[11]. Twins appeared in many grains after first few cycles. Increase in cycle number leads to increase in twinned layers which after 50 cycles occupy a great part of grain, and the inherent slip lines were observed in twins.

The tin twinning during LTC was studied in[13,19,23] and that of Zinc in[21]. A great tendency to twinning reveal magnesium polycrystals[16] as well as number of Fe–Cr–Ni steels under external stress at cooling to helium temperatures[39].

Twinning during LTC results not only in the thermal stress relaxation, but is a more effective factor in grain grinding compared to slip. Moreover, in some cases twinning decreases the macroscopic anisotropy of physico-mechanical properties of textured metals and alloys. Thus, e.g., in polycrystalline beryllium the LTC under load in the range 77 $\leftrightarrows$ 300 K leads to intensive twinning[40] in large volumes of initial grains and results in a considerable decrease of the latter. Statistical analysis
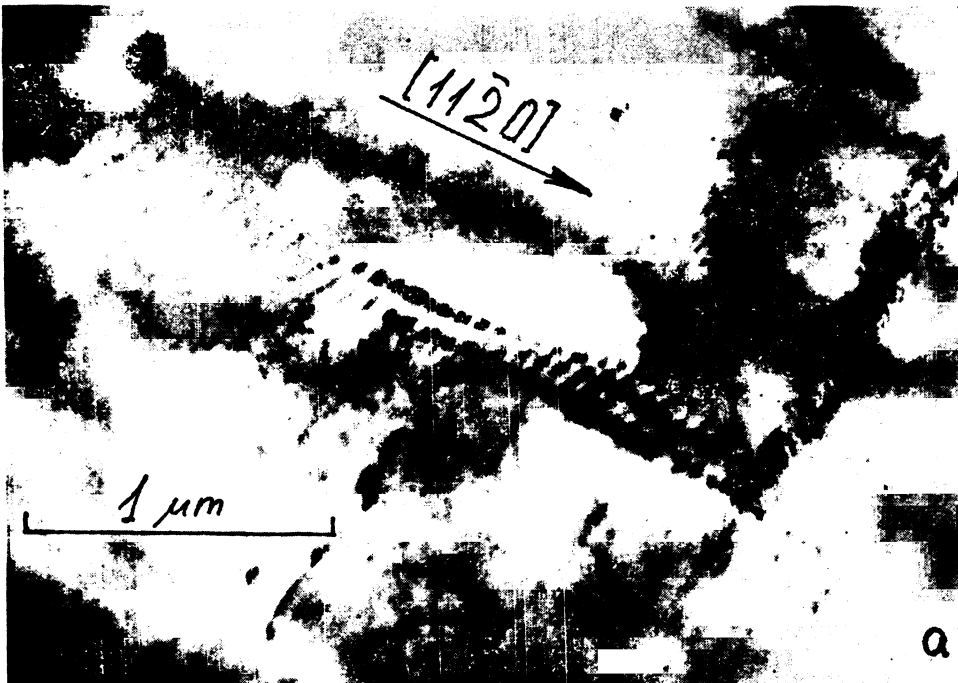

Fig. 1. Helical dislocations (a) and elongated loops (b) in Ti + 5%Al alloy after LTC.
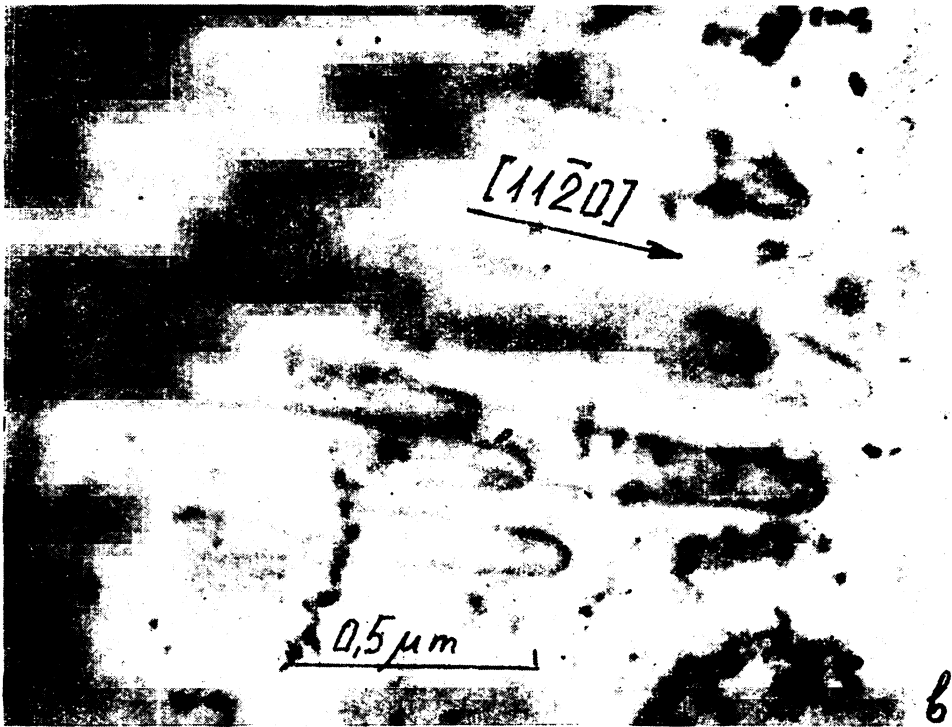
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Fig. 1b.

of the metallographic photos and x-ray investigations showed, that after LTC the mean grain value (about 50 μm) decreased by a factor of two (about 20 μm).

Change in structural state due to phase transformations

During LTC the structural state of metals and alloys may change due to different phase transformations (occurring parallel with slip and twinning), among which the polymorphic transitions have the greatest influence on structural state and physical properties of materials. The polymorphic phase transformations affect the structural state of metals and alloys similar to twinning, since in both cases the interface and reoriented crystal lattice regions appear in the initial grain volume, and this also leads to grain grinding. From twinning it differs by that the crystal lattice of new phase by polymorphic transformations differs from the initial one involving change in volume and shape of the region transformed. This property is inherent many pure metals and alloys such as uranium, cobalt, iron, lithium, titanium, manganese, tin, iron–chromium–nickel, copper–aluminium, silver–zinc aloys etc.[12,41−48]. Different volumes of regions of initial and transformed phases give

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rise to internal stresses promoting residual deformations in samples. The residual deformation value depends on alloy composition, depth and kind of transformation, alloy structural state, cycling parameters and may achieve a great one.
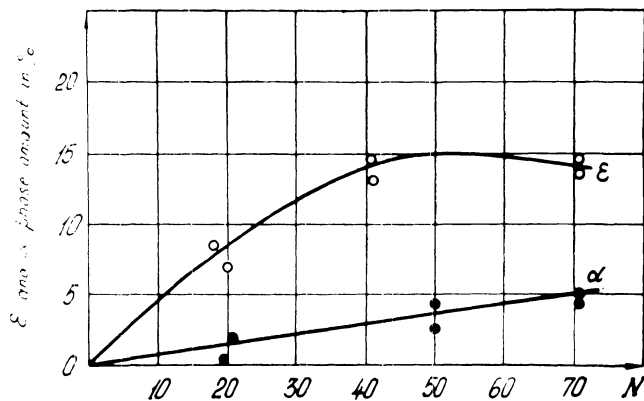

Fig. 2. Dependence of ε- and α-phase amounts on cycle number for I Cr10Ni steel samples.
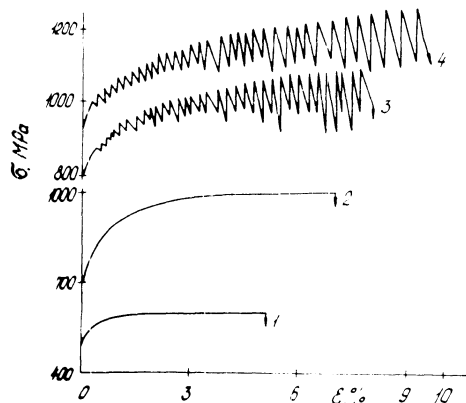

Fig. 3. Work hardening curves for 3M alloy at 300 (1), 77(2), 4 2 (3), 4 2 (4) after 300 cycles under load.

Role of the above factors during LTC is now studied well[12]. However, there are few investigations of LTC effect on phase transformations what makes it often impossible to identify causes of different results in LTC and HTC for identical alloys. Only a few papers are known about the influence of martensite transformations during LTC in the temperature tange 20 to 77 K on structure and properties of some structural steels[49-52]. At the same time time the martensite transformations taking

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place in the low-temperature range without load or static loading were studied more than once.

Important results on the structural state change in 18Cr8Ni and 18Cr10NiT alloys during LTC were obtained in Ref.[53-57]. Using metallographic, x-ray and magnetometric techniques it was shown that in 18Cr8Ni steel α-martensite forms in initial austenic γ state during LTC in the range 300 $\rightleftarrows$ 77 K. This leads to new interfaces in initial grains and consequently to their grinding. It was established that with the increase of cycle number, N, the grain size, d, decreased from $d = 15\ \mu m$ in the initial state to $d = 4\ \mu m$ after 15 cycles.

The structural changes in 18Cr10Ni steel during LTC are qualitatively and quantitatively different from those in 18Cr8Ni steel. This is due to the higher nickel content and consequently to the higher stability of initial austenite.
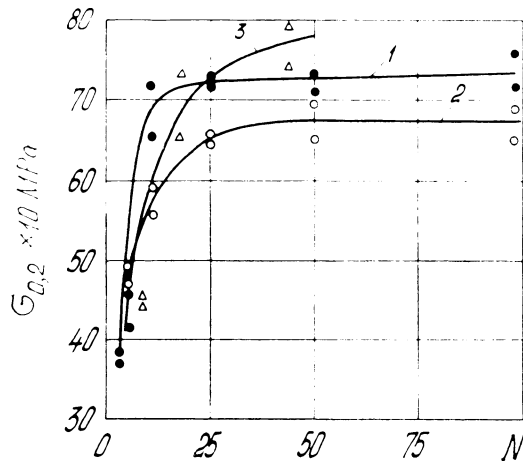

Fig. 4. Dependence of yield stress on cycle number for 18Cr Ni alloy at 77 K. (1) cycling in the range 300 $\rightleftarrows$ 77 K at σ_n 0.8$\sigma_{0.2}$; (2) cycling in the range 300 $\rightleftarrows$ 77 K at σ_n 0; (3) cycling in the range 300 $\rightleftarrows$ 472 K at σ_n 0.8$\sigma_{0.2}$.

In 18Cr18Ni alloy LTC leads to α martensite formation and this has a considerable influence on mechanical properties. Figure 4 shows dependences of yield strength for this steel at 77 K on cycle number in different ranges of LTC. At cycling in the range 300 $\rightleftarrows$ 77 K the most substantial growth of $\sigma_{0.2}$ (by about of factor 2: from 360 MPa in the initial state to 75 MPa after cycling) occurs already after a few first cycles. Then (after about 20−25 cycles) increase in $\sigma_{0.2}$ is practically stopped (curve 1). Without external load (curve 2) change of yield stress with cycle number is smoother, the maximum $\sigma_{0.2}$ value is achieved at the higher cycle number and it is markedly lower than during cycling under load. During cycling in the range 300 $\rightleftarrows$ $\rightleftarrows$ 4.2 K yield stress increases with the cycle number (curve 3), and after $N = 50$ it

does not yet reach maximum value, indicating that process of the structural state change is not completed.

In 18Cr10Ni steel, where LTC under load $\sigma_n = 0.8\,\sigma_{0,2}$ at first involves $\gamma \rightarrow \varepsilon$ and then $\varepsilon \rightarrow \alpha$ transformation, the phase transitions also lead to a considerable change in mechanical properties (Fig. 5). Fig. 5 shows N dependences of $\sigma_{0,2}$,
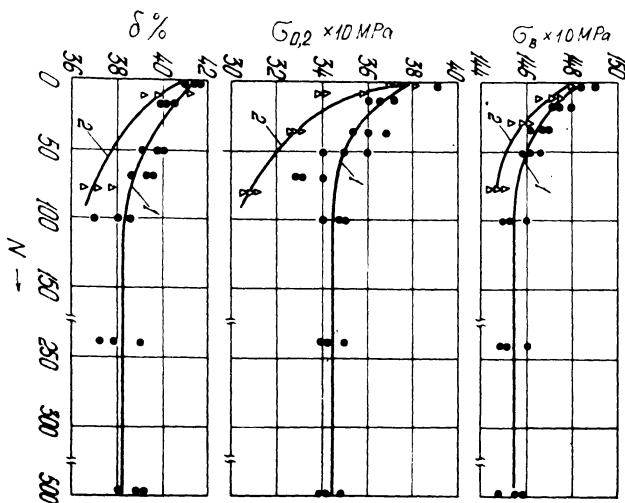

Fig. 5. Dependence of the ultimate resistance, yield stress and relative elongation on cycle number for 1 Cr10Ni steel at 77 K and the load $\sigma_n = 0.8\sigma_{0,2}$: (1) cycling in the range 300 $\rightleftarrows$ 77 K; (2) cycling in the range 300 $\rightleftarrows$ 4 2 K.

σ_B and δ at 77 K for the samples cycled in the ranges 300 $\leftrightarrows$ 77 K and 300 $\rightleftarrows$ 4.2 K. Cycling in liquid helium has a greater influence than cycling in liquid nitrogen, what is due to higher thermal stresses appearing during LTC in the range 300 $\rightleftarrows$ 4.2 K compared to the range 300 $\rightleftarrows$ 77 K.

LTC of 18Cr10Ni steel leads to the qualitatively new character of temperature dependence of $\sigma_{0,2}$ compared to initial samples and 18Cr8Ni steel. Figure 6 shows N dependence of $\sigma_{0,2}$ for this steel at 77 K and 300 K. With the N increase $\sigma_{0,2}$ at 77 K (Fig. 5) decreases from 400 MPa at $N = 0$ to 330 MPa at $N = 70$. On the contrary, $\sigma_{0,2}$ at 300 K with the N increase changing from 22 MPa at $N = 0$ to 32 MPa at $N = 70$. At $T = 4.2$ K $\sigma_{0,2}$ behaves similar to $\sigma_{0,2}$ (N) at 77 K.

Analysis of the kinetics of origination and developing ε and α phases at yield stress at 77 K and 300 K showed [57, 59] that at 300 K effect of hardening is explained in the same way as for 18Cr8Ni alloy by the decrease in the effective grain size d due to new interfaces appearing as lamellae of ε phase. Here the Hall-Petch relationship

$$\sigma_T = \sigma_0 + Kd^{-1/2} \tag{2}$$

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is observed where σ_0 is the deformation resistance of crystal without grain boundaries (Fig. 7). However, parameters of the equation (2) for 18Cr10Ni alloy greatly differ from those for 18Cr8Ni steel $\sigma_0 = 25$ MPa and $K3 = 22.6$ MPa for 18Cr10Ni steel,
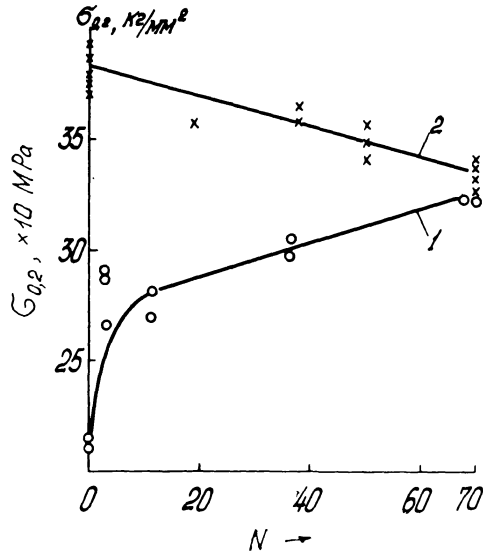

Fig. 6. Dependence of $\sigma_{0.2}$ on d in $\sigma_{0.2} - d^{-1/2}$ coordinates. (1) 18Cr8Ni; (2) 18Cr10NiT steels.
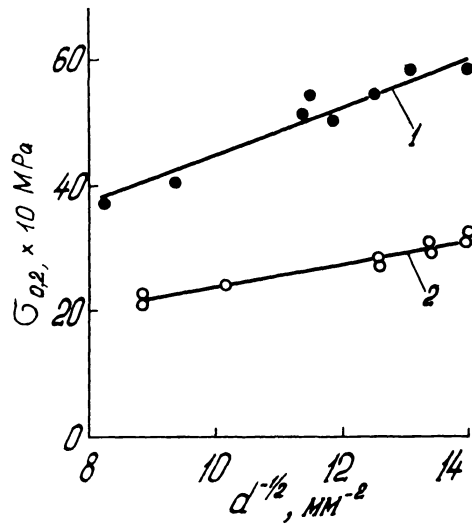

Fig. 7. Dependence of $\sigma_{0.2}$ on N: (1) at 300 K; (2) at 77 K; for 18Cr10NiT steels.

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and $\sigma_0 = 150$ MPa and $K = 33$ MPa for 18Cr8Ni steel. Comparison of these values shows that the plastic deformation resistance in 18Cr8Ni steel (both in grain and over the boundary) is greater than for 18Cr10Ni steel.

Analysis of the structure of 18Cr10Ni steel samples strained to $\sigma_{0,2}$ at 300 K shows the practical invariability in quality and quantity of the phases appearing during cycling. On the contrary, straining to $\sigma_{0,2}$ at 77 K leads to an essential development of ε-phase during cycling increasing length and width of its lammellae. Moreover, the α phase appears forming on ε-phase lammellae. The ε-phase development at yield strength at 77 K seems to be due to decrease in the stacking fault energy (ε-phase nucleus) in 18Cr10Ni steel with temperature decrease as has been shown in Ref. [60]. These results allow to make an important conclusion that the softening effect in 18Cr10Ni steel at 77 K and 4.2 K with N increase is associated with the appearance of α-martensite of bcc lattice during straining forming out of ε martensite of hcp lattice and with the considerable development of the ε martensite already available. Thus, with increasing N the ε-phase increases and the number of places increases where α-phase can form. Appearance of α-phase due to its shear nature will favour beginning and increase of plastic deformation rate, giving rise to an essential decrease in the yied stress measured. Hence the anomaly in temperature dependence of $\sigma_{0,2}$ for 18Cr10Ni type steeels after cycling becomes clear.

Conclusions

The brief enumeration and analysis of the data available show that LTC of structural metals and alloys of different lattice types may involve essential changes in their structural state and mechanical properties. The LTC effect depends on many external and internal factors whose optimal choice permits considerable improvement of material mechanical properties, as has been shown on Ti + 5%Al alloys, Be and stainless steels.

LTC seems to be an effective technique creating the reserve of mechanical properties in metals and alloys in the low temperature range thus allowing to decrease metal consumption in cryogenic structures.

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