EFFECT OF STRUCTURE OF NICKEL ALLOYS ON THEIR MAGNETIC PERMEABILITY VALUE

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It is known that at heating of ageing type invar decomposition of supersaturated solid solution take place. That stipulated increase of hardness of the material [1]. At cooling invars with metastable austenitic in liquid nitrogen in them hardness increase too at the expense of crystals of martensite arise [1]. The purpose of the paper is investigation of correlation between hardness and structure change and magnetic permeability. That correlation is going to be possible nondestructive testing thermal treatment of invars.

Model alloy N30K10T3 content in mass % : 0.01C; 30.0Ni; 10.0Co; 3.2Ti and Fe for balance. An ingot weighing 10 kg was melted in a vacuum induction furnace from pure components and forge at 1100^0C in to a different cross-section rods. Preliminary treatment conclude isothermal holding at 1150^0C in the course of 2 hours and following quenching in water. Invar N30K10T3 after quenching at 1150^0 C in water has ferromagnetic structure. Austenite (γ-phase) of the alloy has Curie temperature $T_c=200^0$C and besides it is metastable at cooling in liquid nitrogen because of it's temperature of start of martensitic transformation is $M_s \approx -80^0$C [1]. Besides, γ-phase present some supersaturated solid solution capable to decomposition at following heating. Magnetic permeability is measured with eddy current method [2,3].

The given alloy may be strengthen by means of next six methods: 1- aging in interval of temperature from 550 to 750^0C; strengthen take place at the expense of precipitation intermetallide phase in austenite [1]; 2-cooling in liquid nitrogen of the material already strengthened with first method; at that martensite of cooling arising in quenched and aged samples as result of $\gamma \rightarrow \alpha$ transformation strengthened the alloy additionally [1]. 3- by means of ageing of the specimens preliminary strengthened with phase-hardened method [1] ; decomposition of supersaturated solid solution in phase-hardened specimens fulfill in the same temperature interval as in the first method; 4- after phase-hardening and aging the specimens is cooled in liquid nitrogen; precipitated martensite of cooling strengthened the alloy additionally; 5- after quenching a cold deformation at 20^0C within 30% and then aging procedure is carried out in the same temperature interval as in first method [4]; strengthen is achieved at the expense of dislocations arise in structure as result of deformation and at the expense of decomposition of supersaturated solid solution; 6- deformed and aged specimens is cooling in liquid nitrogen for part of deformed austenite transformation in martensite that strengthen invar additionally .

The strengthened specimens were used for measurement: 1 –hardnes with Vickers instrument and standard strategy; 2- initial magnetic permeability [2,3] -- with installation which structure circuit is shown at Fig.1. Oscillator 1 create sinusoidal voltage that increase in amplifier 2 then enter coil 3 of solenoid. Measured sample 5 inserted in coil 4 situated in solenoid 3 in which homogeneous alternated field is created. Voltmeter 7 measured the magnitude of signal at coil 4. Other instrument 8 is for frequency measurement. The phase meter is for phase of signal of coil 3 measuring. Resistor 6 is for reference signal. The installation work as follow: at given frequency the readings of voltmeter 7 and phase meter 9 takes into account. These data is monitored according strategy [2,3]. The calculations result in the magnetic permeability value.

Quenched rods were subjected to 30% deformation at 20^0C in brook type of rolling stones. The structure of specimens we studied with optic (NEOPHOT-2) and electronic (JEM-200 CX) microscopes. Before the microstructure researches begin we etched polished section in electrolyte consisting of 90 parts of perchloric acid and 10 parts of acetic acid with subsequent finishing etching in a 4% solution of nitric acid in ethyl alcohol. A foils for electron microscope we use electrolyte consisting of 800 ml ortophosfor acid and 170 g of chrome angydride.

The method so-called phase-transformation-induced hardening (phase-hardening) has been developed. It is schematically illustrated in Fig.2 [4]. The enhancement of strength characteristics of austenitic alloys using this method is based on the effect of strengthening of γ-phase as result of sequentially realization of forward and reverse martensitic $\gamma \rightarrow \alpha \rightarrow \gamma$ transformations. Let us to consider Fig.2 in some more details. At room temperature (at point 1 in Fig.2) we have initial unstrengthened austenite. The forward martensitic transformation $\gamma \rightarrow \alpha$ (point 2 in Fig.2) is carried out in the process of steel cooling to below the M_s temperature in liquid nitrogen or in special cooling chamber. Then the cooled sample is warmed up to room temperature (point 3 in Fig.2). The resulting two-phase $(\gamma+\alpha)$ alloy is heated to a temperature above the range of a reverse martensitic transformation $(A_s - A_f)$ (point 4 in Fig.2), where A_s and A_f - are the temperatures of start and finish of reverse transformation. This heating results in the reverse $\alpha \rightarrow \gamma_{ph}$ transformation which is accompanied by a formation of the so-called phase-hardened austenite γ_{ph}. Then the sample is cooled to room temperature (point 5 in Fig.2). In this case , the alloy microstructure contains strengthened γ_{ph} phase and retained austenite γ_{ret} (which did not take part in the cycle of the $\gamma \rightarrow \alpha \rightarrow \gamma_{ph}$ transformation). The phase-hardened austenite is stable at room temperature, since the M_s temperature of the alloy lies in the range of negative ones. The number of sequential cycles of martensitic $\gamma \rightarrow \alpha \rightarrow \gamma$ transformations can be increased at request.

Structure of an alloy

The size of grains in N30K10T3 invar after it's quenching is 300-350 mkm in average. In spite of that austenite of the studied alloy is a super saturated solid solution, an aging of it is not take place till temperature $T_a = 500^0C$. There is known [5] that in austenitic alloys of Fe-Ni-Ti system decomposition of supersaturated solid solution proceed in two mechanism: continuous and discontinuous one. In first case precipitation of γ'-phase particles (chemical composition Ni_3Ti) with an **fcc** lattice isomorphous to the austenite matrix. The particles of γ'-phase precipitate simultaneously in whole volume but the velocity changes from one area to the other one.

In second case the particles of η-phase of the same chemical consistence as in first case is precipitated. That phase has a hexagonal structure. Discontinuous decomposition present plane shape of colonies (cells) like those of perlitic one in construction low-alloy steels after quenching and abating. The sells consist of alternated direct and parallel plates of η-phase and austenite (γ-phase). That γ-phase is depleted about nickel and titanum in compare with quenched state. In temperature interval within $550 – 650^0C$ a decomposing of supersaturated solid solution proceeds only by first mechanism [5].

After aging at $T_a = 700^0C$ besides continuous decomposition initial study of discontinuous one is observed as well. That display in increase of a thickness of grain borders at observe in optic microscope. The temperature interval in which discontinuous decomposition in solid solution still take place is 200^0C. It begins from $T_a = 700^0C$ and finishes at $T_a = 900^0C$. At that the maximum quantity of decomposed particles is observing after aging at $T_a = 800^0C$.

At electron-microscope investigation in austenite grains of quenched invar rare plates of martensitic phase is observed (Fig.3a). Presence in the alloy abovementioned α-phase at room temperature testifies that in quenched alloy N30K10T3 proceeds weakly expressed isothermal $\gamma \rightarrow \alpha$ transformation [1]. At cooling invar till the temperature $T_{cool} \leq -80^0C$ begin to realize a clear expressed athermal martensitic transformation ($\gamma \rightarrow \alpha$).

Cooling quenched sample in liquid nitrogen lead to formation into γ-phase a martensitic crystals of α-phase athermal type (Fig.3b) [1]. The crystals of that type is forming in alloy when aged specimens is cooling in a liquid nitrogen.

If quenched invar to strengthen with phase-hardened method then isothermal martensite that has been formed at quenching just vanish. That take place because martensitic crystals at heating till 800^0C undergo to reverse transformation $\alpha \rightarrow \gamma_{ph}$ .Analogous process take place at heating athermal martensite which forms in alloy when quenched martensite is cooling in liquid nitrogen. The crystals of phase-hardened austenite (γ_{ph}– phase) is observed as black needles upon white background of retained (that did not take part in $\gamma \rightarrow \alpha \rightarrow \gamma_{ph}$ transformation) austenite (Fig.3 c) [1].

In structure of invar strengthened with phase-hardened method was observed separate thin crystals of athermal martensite in electron microscope (fig.3d) . Their existence apparently stipulates as following [1]. In reverse martensite

transformation $\alpha \rightarrow \gamma_{ph}$ austenite subjected to aging and γ'– phase precipitates. Precipitation of intermetallic particles depletes the matrix of nickel and titanium and the temperature of start of martensitic transformation becomes higher of the room one. That strengthened with phase-hardened method material at cooling from $T_a = 800^0$C till room one suffer to martensite $\gamma \rightarrow \alpha_a$ transformation. Thanks to dispersion of crystals of α_a-phase they is not visible in optic microscope.

Study the aging phase-hardened specimens with optic microscope allows the conclusions as follow: 1) the higher the temperature of aging the worse visible a crystals of phase-hardened austenite; one can explain that as result of intermetallide particles appearing inside γ_{ph}- phase; 2) after aging at temperature higher than 600^0C in retained austenite appears α_a-phase of athermal type. Again the higher temperature of aging the worse visibility of the martensitic needles.

Structure of deformed alloy was studied with optic and electron microscopes. In first case a presence of martensite of deformation has not been founded. In second case separate disperse crystals of martensite of deformation has been observed (Fig.3e) [1]. Besides in γ-phase one could see a dislocation formed a strip banded structure (Fig.3f) [1]. This dislocations decorate with intermetallid phase at aging process and as result that strip banded structure one can observe not only in electron microscope but in optic as well (Fig.3g) [1].

Thus, structure of deformed invar consist of austenite having big density of dislocations that is decorated with intermetallid particles, of martensite of deformations (α_d) and deformed isothermal martensite (α_{is}) appeared in quenched process.

Decomposition of supersaturated solid solution at temperature $T_a \geq 650^0$C make austenite less stable and as result of that at cooling the alloy from aging temperature till room one take place martensitic transformation $\gamma \rightarrow \alpha_a$. Inside of austenite grains a crystals of martensite of aging of athermal type (α_a –phase) is appearing (Fig.3h) [1]. At temperature of aging increase till $T_a = 700^0$C a borders of grains become more thick. That is testified that at shown temperature of heating the discontinuous mechanism of decomposition of supersaturated solid solution become more clear. Sells of discontinuous decomposition is not visible in optic microscope, but they visible enough in electron one.

Cooling of deformed and aged specimens in liquid nitrogen stipulates partial transformation of γ-phase in martensite of cooling ($\gamma \rightarrow \alpha$) athermal type. In structure of deformed specimens aged at $T_a \geq 650^0$C besides of austenite still two martensite present: martensite of cooling (α) and martensite of aging (α_a).

Hardness of the alloy

After quenching a hardness of invar is HV=190, and it is not change at tempersture of aging till $T_a = 500^0$C (Fig.4a, curve 1) because decomposition of supersaturated solid solution at these value of temperature is not take place. At first variant of the treatment, heating within interval of temperature from 600 to 750^0C make it possible to increase a hardness of the material. A hardness

enhance is stipulated by arising of intermetallid particles in austenite and crystals of martensite of aging appearing in matrix (α_a) as well.

Cooling of the quenched specimen in liquid nitrogen has been stipulated in it $\gamma \rightarrow \alpha$ transformation. Second method of treatment is providing additional growth of durability of whole aged specimens (compare the curves 1 and 2, Fig.4a). Nevertheless the form of curves of dependences HV(T_a) in both variants is the same (compare the curves 1 and 2, Fig.4a).

After strengthen of quenched invar by means of phase-hardened ($\gamma \rightarrow \alpha \rightarrow \gamma_{ph}$) it's durability enhance (Fig.4, the curve 3). Within interval of heating from room one till 400^0C the durability level of phase-hardened alloy is not change, because decomposition of supersaturated solid solution at shown range of temperature is not take place. But still at $T_a \geq 500^0$C hardness increase is observed. Compare the curves 1 and 3 (fig.4a) one may to take conclusion that cycle of transformation $\gamma \rightarrow \alpha \rightarrow \gamma_{ph}$ accelerates process of aging. At temperature of aging $T_a = 700^0$C in quenched and phase-hardened alloy too proceeds process over-aging, therefore compare of invar hardness after shown treatments ($T_a = 700^0$C) is not constructive.

Cooling in liquid nitrogen of phase-hardened and aged specimens increases their hardness (compare the curves 3 and 4, fig.4a). At that a hardness rests practically invariable within of interval from room temperature till $T_a = 500^0$C. After achievement of that value the hardness is enhanced and achieved of maximum at $T_a = 700^0$C.

Plastic deformation (30%) of quenched invar increase it's hardness (compare the curves 1 and 5 Fig.4a). As one can see (the curve 5, Fig.4a) heating of deformed γ-phase till $T_a = 400^0$C is not change it's hardness which is increased only at aging temperatures $T_a \geq 550^0$C. If compare the dependence HV(T_a) for not deformed and deformed material (the curves 1 and 5, fig.4a) one may to make conclusion about intensification of decomposition of supersaturated solid solution in last case. An acceleration of aging decrease till zero in case of the alloy heating till $T_a = 700^0$C which is stipulated by proceeding of process of over aging of solid solution.

The sixth method of strengthening (cold deformation+ aging+ liquid nitrogen) is not change the dependence HV(T_a) in compare with fifth one (compare the curve 5 and 6 Fig.4a) but enhances all values of hardness of the deformed and aged material. The enhance of durability is stipulated by transformation at cooling of the part of austenite into martensite ($\gamma \rightarrow \alpha$).

Hardness changes not only because of the temperature changing but because of aging duration too. After $T_a = 600^0$C durability monotonically enhance in the course of time (the curve 1 Fig.5a). However, already after isothermal holding $\tau_a = 2$ h a hardness enhance practically ceases. Monotonous enhance of hardness breaks of already at $T_a = 700^0$C and $\tau_a > 1$ h. Diminish of hardness take place because of processes over aging. The process is displayed, because at the borders of grains discontinuous decomposition begin to develop and γ'-particles become more large.

After first and second variants of treatment of quenched alloy the dependencies HV(τ_a) is analogical (compare the curves 1 and 2 Fig.5a). The difference is that at last case the hardness is higher because of additional appearing of martensite of cooling.

If isothermal holding increase hardness of invar strengthened with phase-hardened method after aging at T_a =600^{0}C monotonically arises (curve 3 Fig.5a), and now it is higher then in case of quenched (not phase-hardened) alloy (compare the curve 1 and 3 Fig.5a). The comparison of these dependences HV(τ_a) allow to confirm that in phase-hardened invar decomposition of super saturated solid solution take place more intensive then in quenched one. At temperature of aging increase from 600 to 700^{0}C now the dependence HV(τ_a) change monotonically, and maximum value of durability achieves at τ_a =1 h. Increase of duration of aging lead to diminish a hardness because over aging process [1]. The maximal level of durability after aging at 600 and 700^{0}C is the same practically.

Now we shall consider the dependence HV(τ_a) for phase-hardened and aged specimens after their cooling in liquid nitrogen. Hardness increase monotonically at T_a= 600^{0}C (the curve 4, fig.5a). The hardness has increased as result of cooling of alloy in liquid nitrogen (compare the curves 3 and 4 Fig.5a). The same dependence at T_a =700^{0}C is changing not monotonically, and the hardness achieves maximal value at τ_a =1 h [1]. In this case the hardness of cooled and not cooled material nearly the same.

As for deformed in 30 % invars at room temperature the dependence HV(τ_a) increase monotonically after aging at T_a = 600^{0}C. In that case after aging in the course of 6 h a durability growth is practically over. However, if aging temperature rise till 700^{0}C the dependence become not monotonically and achieves the maximum at τ_a = 1 h. Increase of duration of isothermal aging (τ_a>1 h) durability diminish because over aging process [1].

After fifth and sixth variants of treatment the dependences HV(τ_a) is analogical (compare the curves 5 and 6 Fig.5a). The difference is that hardness value is higher in last case. It is stipulated by a cooling of alloy in which additionally appear martensite of cooling.

Measuring of magnetic permeability

As above said, all kind of transformations demand the correspondent means to determine them. Optic and electron microscopes the researchers use as traditional instrument, but at as much of directions of researches a considerable help the nondestructive instruments render as well. For this purpose we had studied dependence of magnetic permeability value for different phase transformation in nickelferous alloy. Measuring of magnetic permeability has been carried out with the circuit, which structure schema is presented in Fig.1 (above mentioned) [2,3].

Fig.4 demonstrates the possibility of determination of hardness change using the results of permeability measuring. One can see that the correlation

between magnetic permeability and hardnes value is really exist, but it begins from temperature of aging $T_a = 500^0C$. Diminish of magnetic permeability is stipulated by two factors: 1- decomposition of supersaturated solid solution and transformation of part of austenite in martensite of aging, which has permeability lessen then austenite. In case of phase-hardened alloy the permeability decrease at $T_a > 600^0C$ and the cause of that is the same.

In case of cold deformation on 30% with growth of aging temperature magnetic permeability diminish as well. Decrease of it stipulated by two factors: 1- appearing in the structure of martensite of aging; 2- more intensive decomposition of supersaturated solid solution.

At cooling in liquid nitrogen of deformed and aged specimens the permeability decrease (compare the curve 5 and 6 Fig.4b). Dependences $\mu(T_a)$ for fifth and sixth variants is analogical, but in last case permeability value lessen. Decrease of μ take place because part of austenite transforms into martensite of cooling.

The value of magnetic permeability of quenched alloy is decreased at holding time increases (the curve 1 Fig.5b). Rise of temperature of aging from 600 till 700^0C lead to additional diminish μ.

For third and forth variants of the treatment (the curves 3 and 4 Fig.5b) is observed the same regularity as for first and second variants.

Increase of holding duration at $T_a = 600^0C$ is not effect on permeability of deformed (30%) (the curve 5 Fig.5b). Isothermal holding of deformed specimen $\tau_a = 6$ h at $T_a = 700^0C$ diminish μ value monotonically [1].

Cooling of deformated and aged specimens in liquid nitrogen diminishes magnetic permeability (compare the curves 5 and 6) as well.

Conclusion

Decomposition of supersaturated solid solution in quenched, phase-hardened and deformated invar increases the hardness but decreases the permeability. That phenomena one may use for the purpose at research. Magnetic permeability is measuring with simple eddy current installation which gives to the researchers additional information about of state of alloy at their investigations. That information has been compared with those received by means of using other instruments such as optic and electron microscopes during many years. The accumulated experience make it possible to determine the condition of alloy at studying of very complex phase transformations in quantitative. For example it is quite possible to determine the decomposition degree in the alloy provided it is known the primary condition of experiment, that is temperature of aging, holding time and other treatments as well. Using their exrperience the researchers often resort to measuring of permeability in their investigations that make their work more easy and economizes the time.

References

1.Sandovskii V.A., Uvarov A.I., Kazantsev V.A., Anufrieva E.I., Filippov Yu.I. Research of decomposition of supersaturated solid solution and martensitic transformation in invar N30K10T3.- Physics of Metals and Metallography.- 2003.-T.96.- Ed.5.- p.67-73.

2. Sandovskii V.A. Account of dependence of electro-dynamic parameters of metals at calculations of through type of eddy currents.- Nondestructive testing.-1965.-№10.-p.32-37.

3. About of dependences of permeability from the frequency at testing with superposed transducers.-Nondestructive testing.-1997.-№1.- p.63-66.

4.Malyshev K.A., Sagaradze V.V., Sorokin I.P. Phase rivet of austenite alloys upon ferrous-nickel foundation. –Moscow: Science, 1982.-260 p.

5.Sagaradze V.V., Uvarov A.I. Strengthen of austenite steels. Moscow: Science.-1989.-270 p.

Text to figures

Fig.1. Structural schema of installation for permeability measuring

Fig.2. Schematic diagram for a treatment of metastable austenite alloys upon phase- hardening.

Fig.3. A structure of H30K10T3 invar after different methods of strengthen:

a- crystals of martensite in quenched sample; dark –field image taken using the $(10T)_\alpha$ reflection, magn. x 100000,

b- water quenching and subsequent cooling of the alloy in liquid nitrogen; magn. x 600,

c- crystals of phase-hardened austenite (γ_{ph}); magn. x200,

d-athermal crystals of α-martensite in phase-hardened invar; dark-field image taken using an α-phase reflection, magn. x 59000.

e- crystals of deformation-induced martensite; dark-field image taken using α-phase reflection, magn.x140000.

f- dislocations in austenite grains, strip banded structure; magn. x 30000.

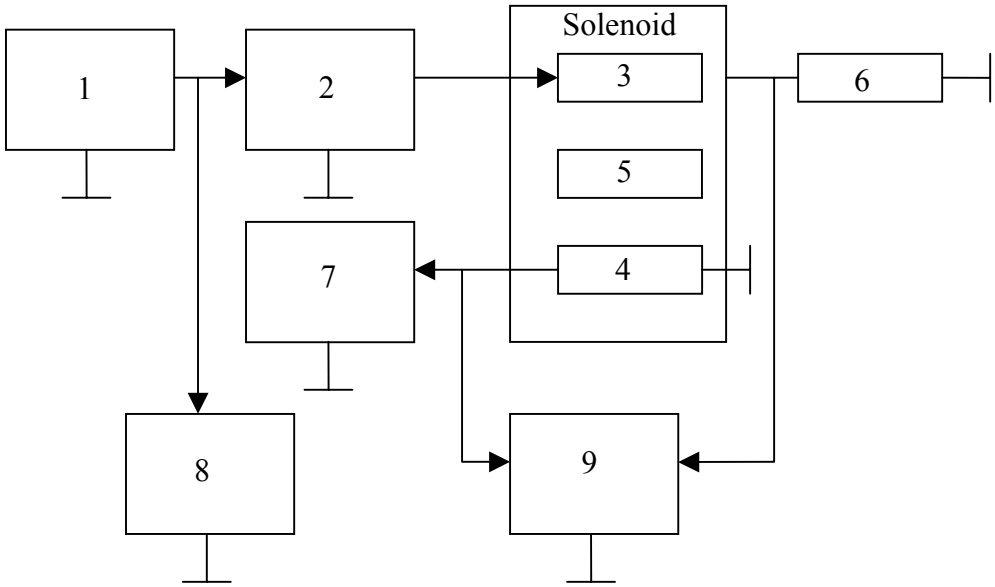
g- strip bands, decorated with intermetallid phase; magn. x 200.

h- aging-induced martensite in aged alloy; magn. x 100.

Fig.4. Variations of hardness HV(a) and magnetic permeability μ(b) after aging at various temperatures T_{ag} with isothermal holding for τ_{ag} =1 h: 1- quenched alloy; 2- alloy subjected to aging and subsequently cooling in liquid nitrogen; 3- phase-hardened and aging; 4- phase-hardened alloy subjected to aging and subsequently cooling in liquid nitrogen; 5- alloy subjected to quenching, deformed to 30% and subjected to aging, 6- alloy subjected to quenching, deformed to 30% , subjected to aging (τ_a=1 h) and subsequently cooling in liquid nitrogen.

Fig.5. Variations of aging holdings, dependence of hardnes (a) and magnetic permeability (b) from duration τ_a at $T_a = 600^0$C for difference variants of treatment:

1- quenching and aging; 2- quenching, aging and cooling in liquid nitrogen;
2- phase rivet and aging; 4- phase rivet, aging and cooling in liquid nitrogen;
5- quenching, cold deformation and aging; 6-quenching, cold deformation, aging (τ_a =1 h) and cooling in liquid nitrogen.



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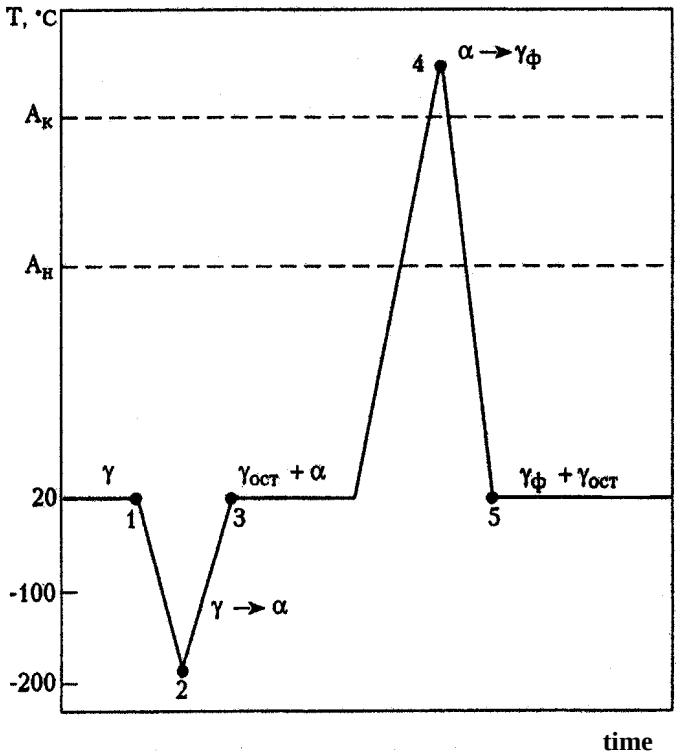

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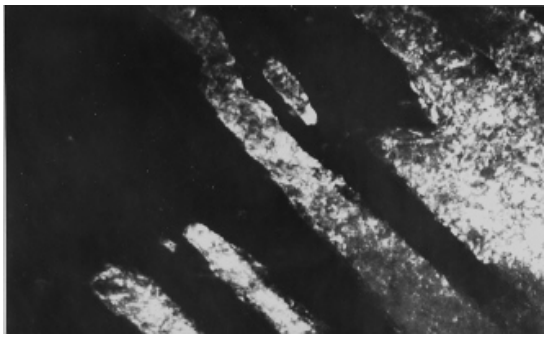

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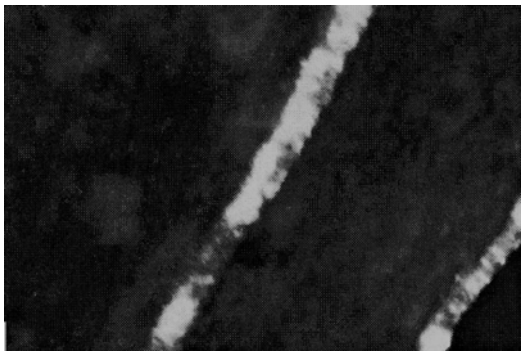

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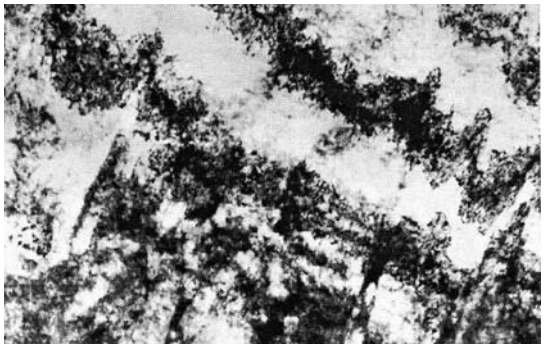

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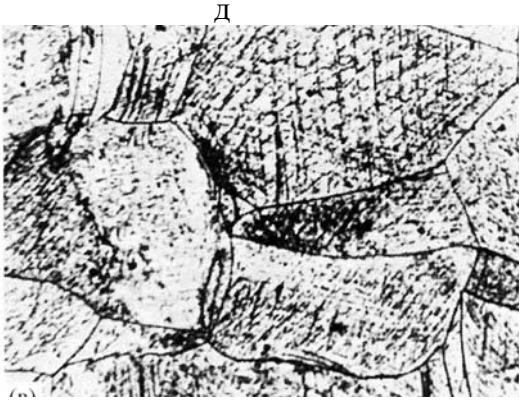

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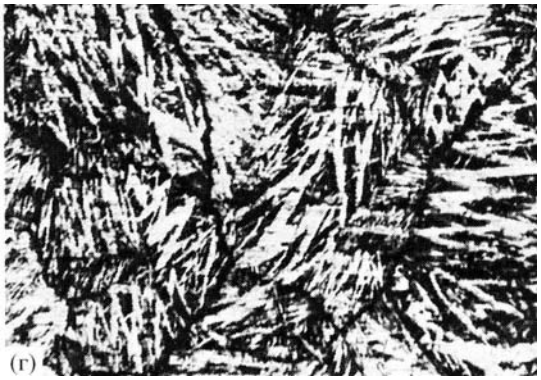

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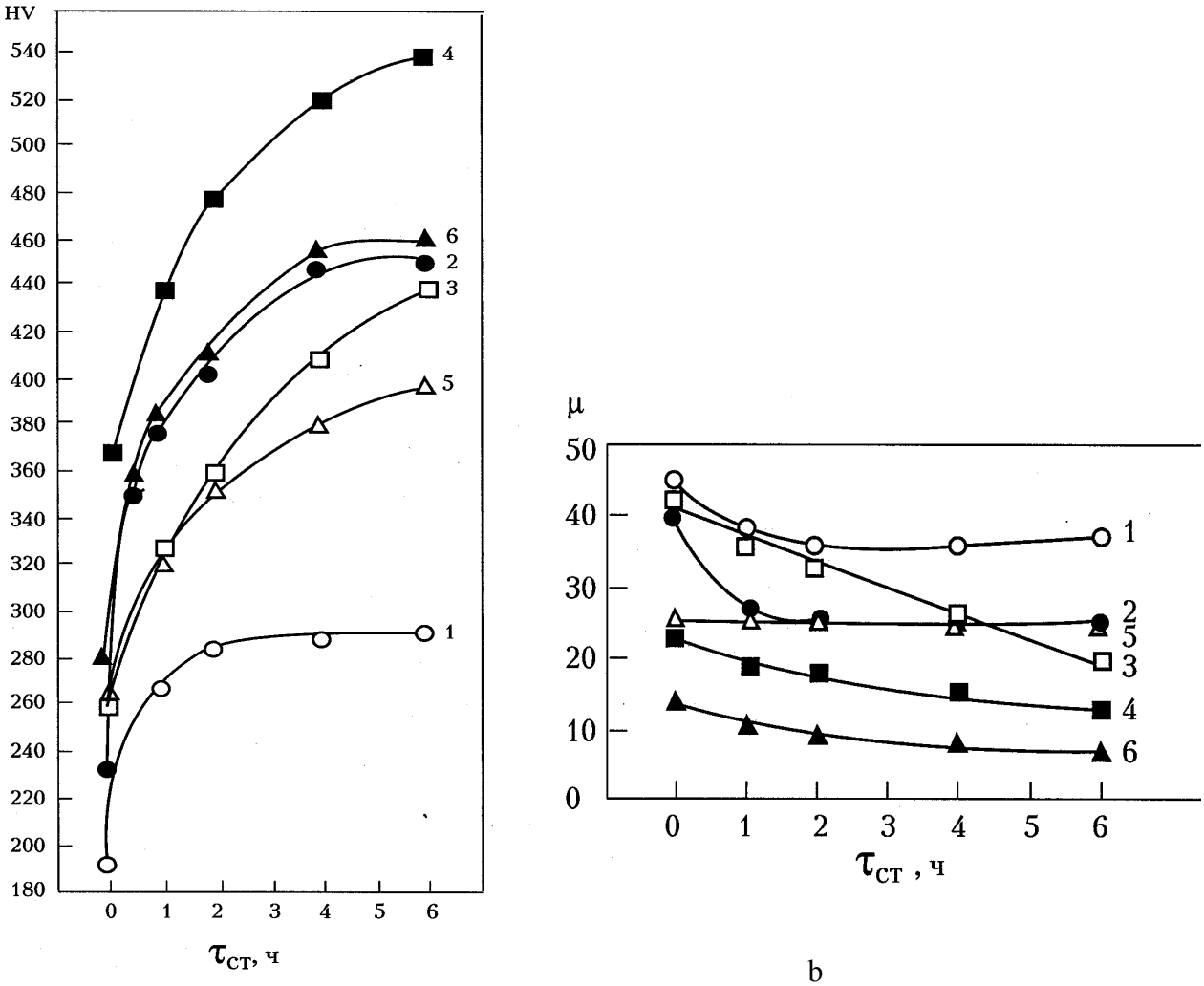
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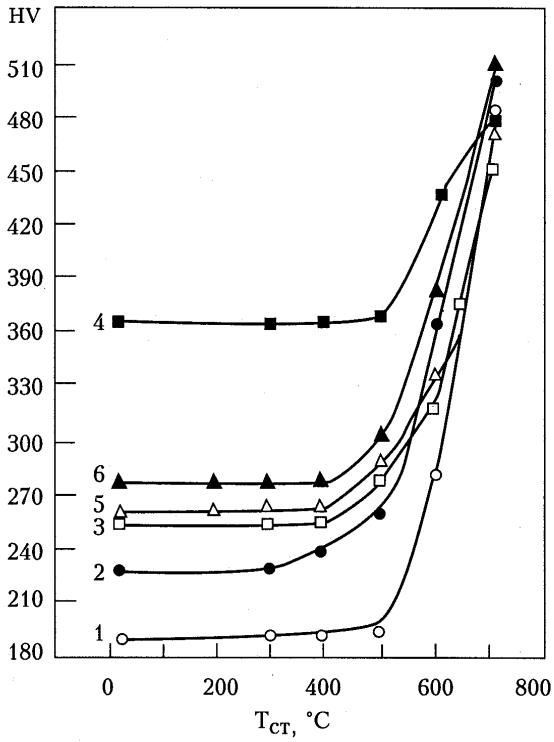
Fig.4.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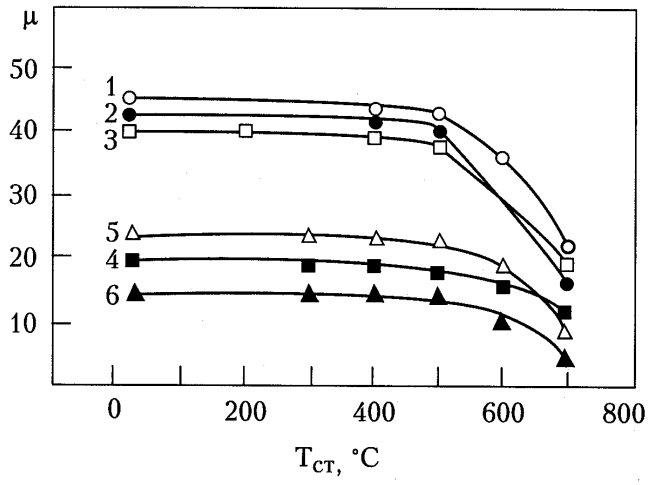
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Fig.5.