presence of the second maximum at thermogram which gives an opportunity of system supplementary reaction. Then the product cools as an inert body.

Comparatively rapid batch cooling at particle size 130 ± 23 mkm gives no opportunity to implement the process of secondary structure formation; plateau appears instead of the second peak, the system has no time to transfer to equilibrium state according to the original stoichiometry.

Conclusion

It follows from the carried out series of experiments that phase composition of the product of SH-synthesis carried out in the mode of thermal explosion in the system Ti+3Al depends on particle size and monophase

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product of stoichiometry TiAl₃ is obtained both on small (in the experiment conditions 55 ± 7 mkm) and large $(180\pm32 \text{ mkm})$ size of titanium particles at induction period termination. At intermediate fraction (130 ± 23) mkm) at induction period termination the product of synthesis is monophase. Similar anomalous effect is explained by the fact that at small particle size the all-burn time is compared with induction time and heat release has no time to influence considerably on phase formation process. Synthesis is finished at the stage of primary structure formation. At large particle size the long diffusion time develops conditions for intensive occurrence of the process of secondary structure formation that is displayed in appearance of the second peak at thermogram. Temperature growth connected with it stimulates formation of the phase corresponding to original stoichiometry.

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CHANGE OF THE SPRING Cr-Ni ALLOY MICROSTRUCTURE AFTER AGEING

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It has been stated that ageing of the tempered alloy 47XHM at temperature 500 °C during 5...10 h does not result in disintegration of oversaturated firm solution, at ageing temperature rise up to 600 °C attributes of disintegration in particles of α -phase of homogeneous type start to be shown. It was shown that after tempered samples ageing at 700 °C the faltering disintegration with allocation of not coherent α -phase on the basis of chrome develops intensively, and its volume fraction increases with increase in ageing time reaching the maximal values in 5...10 h of ageing.

As it is known [1] owing to high elastic properties the precipitation hardening alloys on Ni-Cr basis are applied at manufacturing elastosensitive elements of various devices and machines. Moreover, alloys Ni-47Cr-Mo are perspective materials for elements of construction of nuclear and thermonuclear reactors [2]. In work [3] it is shown that radiation resistance of alloy 47XHM may be considerably improved changing the initial structural-phase state.

Investigations [4] of hardened alloy 47XHM show that sample phase-structural state is influenced by hardening temperature, cure time for hardening and cooling rate. In work [5] peculiarities of change of mechanical properties of alloy 47XHM depending on hardening modes are detected and hardening optimal conditions are determined. The aim of the given paper is to study the change of microstructure of spring alloy 47XHM at aging in temperature range 500...700 °C.

Material and investigation technique

Precipitation hardening alloy 47XHM of standard chemical composition (47 % – Cr, 5 % – Mo, res. – Ni) is selected as research material in this paper.

Carrying out heat treatment of alloy 47XHM, heating for hardening from 1250 °C during 1...2 min and aging in temperature range 500...700 °C were carried out in laboratory electric-tube furnace of resistance of the type SUOL-0,4.4/12-M2-U4.2 in vacuum with residual pressure not more than 1 Pa. Sample state was recorded by hardening in cold water. Structure-phase state of samples was studied by optical (NEOPHOT-21, MIM-7) and electron (EM-125K) microscopes.

Microsections for metallurgical study were polished and etched by electrolytic method in 10 % acetic-chloric electrolyte. Grain size, mechanisms of extraction and phase volume fraction, presence of twins and other defects were controlled by microstructure.

Samples for electron microscopy in the form of discs were prepared by the method of stream electropolishing as well as the method of foil thinning.

Results and their discussion

Metallographic and electron-microscopic investigations of samples aged in temperature range 500...600 °C showed (Fig. 1 and 2) that complex processes of formation of short-range ordering, layering and initial stages of disintegration reflected on selected area diffraction pattern (Fig. 2, c) occur in alloy.

After aging at 500 °C during 5...10 h alloy structure does not practically differ from hardened alloy structure (Fig. 1). Grain bounds are well decorated by particles of α -phase and small particles, probably, chromium or molybdenum carbide. Inside the grains as well as in the case of hardened samples a great number of annealing twins is observed. They usually look like bands limited by parallel lines connected with planes coherent to them {111}.

If in hardened alloy aged at 500 °C twins do not often cross all grains but terminate inside the grain then at 600 °C of aging ingrained twins are completely absent in alloy structure (Fig. 2, a). Typical feature of grain boundary after specified heat treatment is absence of their curvature and as a result structure stability and equiaxity. But on some areas structure inhomogeneity (very high content of small particles of α -phase) caused, obviously, by alloy pouring defects (Fig. 2, *a*) is observed.

In microstructure of samples aged at 600 °C some changes in comparison with samples aged at 500 °C are observed. First of all it concerns structural changes in particles of α -phase (Fig. 2, b) in which disintegration of oversaturated solid solution, probably, of zone type is appeared more precisely than at 500 °C. At point micro electron-diffraction pattern taken from large particles of α -phase long tension bars passing both through lateral reflections and through central spot appear. It indicates not only formation of lamellar extractions but the presence of great deformation. In γ -matrix of alloy in studied aging temperature range no indication of disintegration is observed.

The results of investigation of structural and phase transformations at aging temperature 700 °C are the most interesting ones. It is connected, to the large extent, with very sharp increase of mechanical properties conditioned by development of discontinuous disintegration. Micros-tructures obtained at optical microscope are given in Fig. 3. Analysis of microstructures shows that at increase of aging time the volume fraction of discontinuous disintegration sharply increases. These data correlate well with the results of mechanical properties of alloy 47XHM undergone dispersion hardening at 700 °C.

It is seen on photos that discontinuous disintegration is conducted by occurrence at grain boundaries of exceed etching which spread deep into the grain at isothermal



Fig. 1. Microstructure of alloy 47XHM after heat treatment by the mode: hardening from 1250 °C, 1 min, aging at 500 °C with different aging: a) 1 h, ×450; b) 10 h, ×8500; c) 10 h, ×8500



Fig. 2. Microstructure of alloy 47XHM after heat treatment by the mode: hardening from 1250 °C, 1 min, aging at 600 °C with different aging: a) 2 h, ×450; b) 10 h, ×6500; c) selected area diffraction pattern of structure in Fig. 2, b



Fig. 3. Optical micrographs of structure of preliminary hardened from 1250 °C, 1 min alloy after aging: a) 700 °C, 1 h, ×1000; b) 700 °C, 100 h, ×1000



Fig. 4. Structure of the aged alloy 47XHM after hardening from 1250 °C, 1 min: a) 700 °C, 1 h, ×10000; b) 700 °C, 3 h, ×20000; c) selected area diffraction pattern to Fig. 5, b



Fig. 5. Structure of the aged alloy 47XHM after hardening from 1250 °C, 1 min: a) and b) 700 °C, 5 h, ×23000; c) 700 °C, 10 h, ×8500

tempering. On other areas of metallographic section microstructure does not change. On photos of discontinuous disintegration areas both dark regions of high etching on light field of undecayed matrix (Fig. 3, a) are appeared, at great increases (Fig. 3, b), regions of discontinuous disintegration represents colonies of alternating plates.

In order to study cell internal structure of discontinuous disintegration the method of electron microscopy was used. It allowed studying the nature of extracted phase, orientation ratios in the aged alloy as well as morphology of different stages of discontinuous disintegration.

Electron micrographs of the aged alloy 47XHM at 700 °C after hardening from 1250 °C during 1 min are given in Fig. 4–6. Fine-structural investigations showed that after aging of the hardened samples at specified temperature the discontinuous disintegration develops intensively with the extraction of noncoherent α -phase on the basis of chromium and its volume fraction increases at aging time growth achieving maximal values for

5...10 h of aging. The analysis of selected area diffraction patterns showed that particles of discontinuous disintegration in the form of lamellar extractions represent noncoherent α -phase on the basis of chromium (solid solution of nickel in chromium). Point electron-diffraction pattern taken from the area of discontinuous disintegration showing complex selected area diffraction pattern from matrix and lamels of discontinuous disintegration is given in Fig. 4, *c*. Two systems of reflections – from matrix of γ -solid solution of fcc structure and α -phase of bcc structure are seen at electrondiffraction pattern.

Discontinuous precipitation of α -phase in alloy 47XHM starts from grain boundaries where new phase nuclei are originally formed. They propagate into typical cells (Fig. 4, *a*) consisting of lamels (or rather plates or rods) of α -phase and stripped solid solution. Cells of discontinuous disintegration have noncoherent boundary with matrix along which diffusion of dissolved



Fig. 6. Structure of alloy 47XHM after hardening from 1250 °C, 1 min and aging at 700 °C, 10 h: a) ×8500; b) ×8000; c) ×13000

components occurs at high speed that determines high speed of discontinuous disintegration. Along the boundary rounding the cell atoms of dissolved element come to lamels of discontinuous disintegration and stimulate their growth. In this case the boundary moves synchronously with growing lamels. Observation of microstructures (Fig. 4–6) shows that lamels in the cells of discontinuous disintegration are not ideally straight and parallel and they seem to diverge following increasing cell diameter. However, interlamel distance is constant owing to plates occurring again.

In alloy 47XHM oversaturated solid solution disintegrates by complex mechanism as the studied alloy in hardened state is two-phase that is consisting of γ -solid solution of chromium in nickel – matrix of alloy with fcc structure and solid solution of nickel in chromium – α -phase with bcc structure.

At alloy aging at 700 °C disintegration of oversaturated solid solution starts in both phases simultaneously. In matrix of alloy on the basis of nickel the discontinuous disintegration occurs with formation of lamels of α -phase and depletion regions of matrix with chro-

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mium between plates of γ -phase. Simultaneously disintegration occurs in the second phase – α -phase and disintegration has the character of homogeneous with extraction of new phase on the basis of nickel.

Conclusion

Aging of the hardened alloy 47XHM at temperature 500 °C during 5...10 h does not result in disintegration of oversaturated solid solution.

At growth of aging temperature to 600 °C disintegration features appear in particles of α -phase of homogeneous type.

It is shown that after aging at 700 °C of hardened samples the discontinuous disintegration develops intensively at extraction of noncoherent α -phase on the basis of chromium and its volume fraction increases at aging time growth achieving maximal values for 5...10 h of aging.

Aging time increase at 700 °C to 10 h results in increase of lamel size of discontinuous disintegration and distances between them.

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