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Abstract

Full Text

Chemistry

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## Study of the Effect of Aging Time on the Formation and Composition of the $\gamma'$ Phase in Alloys of the Ni–Cr–W–Al–Ti System

*(Presented by Academician I. I. Chernyayev, May 30, 1960)*

The  $\gamma'$  phase, precipitated during decomposition of the primary solid solution, is the phase that determines, chiefly, the high properties of a number of nickel-base alloys. According to data from many authors, the  $\gamma'$  phase is a solid solution based on the compound  $\text{Ni}_3\text{Al}$ , in which, along with titanium, other elements entering into the composition of the alloy may also dissolve (<sup>1,2</sup>). Crystals of the  $\gamma'$  phase have a face-centered cubic lattice, the parameter of which depends on the composition of the alloy. The lattice parameter of the pure compound  $\text{Ni}_3\text{Al}$  is equal to 3560 kX.

There are indications in the literature that in some alloys the  $\gamma'$  phase is metastable and, during prolonged aging, transforms into the  $\text{Ni}_3\text{Ti}$  phase. N. I. Blok and co-workers write about such a transformation in alloys of the Ni–Cr–Al–Ti system (<sup>3</sup>). According to A. Ya. Snetkov, the  $\gamma'$  phase is metastable in alloys that contain a large amount of titanium. The alloys studied, prepared by L. I. Pryakhina and O. V. Ozhimkova, after being held for 134 hr at 1200°, were quenched from 1200° in water and then aged at 900°. The aging time was from 0 to 10,000 hr.

According to microstructural analysis carried out by L. I. Pryakhina and O. V. Ozhimkova, the alloys studied are two-phase and consist of a  $\gamma$  solid solution and the  $\gamma'$  phase. With increasing aging time, in addition to precipitates of rounded form, precipitates in the form of striations appear on the polished section. This may be explained either by a change in the shape of the precipitating phase or by transformation of the  $\gamma'$  phase into the  $\text{Ni}_3\text{Ti}$  phase.

To study the composition and structure of the phases formed in the alloys under investigation, along with other methods, a special investigation was undertaken based on the electrolytic isolation of excess phases. To isolate the  $\gamma'$  phase from the alloys, an electrolyte previously developed by us (<sup>4</sup>) was used, consisting of 50 ml  $\text{HNO}_3$ , 20 ml  $\text{HClO}_4$  per 1000 ml of water. The isolated anodic powders were subjected to chemical and X-ray structural analyses. A weighed portion of powder was dissolved in aqua regia, after which tungsten was separated hydrolytically. After separation of tungsten, the filtrate was transferred

to a volumetric flask, and aluminum, nickel, and chromium were determined from aliquot portions.

To determine small amounts of chromium in the presence of clearly predominating amounts of other elements, it became necessary to verify chemically the possibility of determining gamma amounts of chromium with diphenylcarbazide under the specified conditions. On the basis of the work carried out, rational conditions for determining chromium were developed. In view of the fact that the large amount of tungsten contained in the anodic powders ( $\sim 20\%$ ) entrains some titanium during precipitation, titanium was determined from a separate weighed portion (0.01 g). To bind tungsten into solution,  $\text{H}_3\text{PO}_4$  (1.70) was added. The results of chemical analysis are given in Table 1.

To study the conditions of electrochemical dissolution of the alloys, we carried out isolation of the excess phase in the electrolyte previously proposed by us.

(5, 6) in an electrolyte consisting of 35 g of citric acid, 5 g of  $(\text{NH}_4)_2\text{SO}_4$ , 15 ml of  $\text{HNO}_3$ , and 1000 ml of water.

The chemical composition of the phase isolated in this electrolyte is also given in Table 1.

### **Table 1**

#### **Chemical composition of the $\gamma'$ -phase**

Experimentation, No.	Aging dura- h	Ni	Al	Ti	Cr	W	Sum of ele- ments	Ni	
								$\frac{\text{Ni}}{\Sigma \text{Al, Ti, Cr}}$	
$\gamma'$ - phase iso- lated in an elec- trolyte con- sist- ing of 50 ml HNO <sub>3</sub> , 20 ml HClO <sub>4</sub> , and 1000 ml of water	8	0	65,20 / 14,58 69,03	6,33 / 4,23	3,26 / 4,23	4,26 / 5,09	20,92 / 7,07	99,97 / 100,00	2,9
	1	100	65,31 / 14,95 69,46	6,46 / 3,64	2,80 / 3,64	3,88 / 4,66	21,50 / 7,30	99,95 / 100,10	3,0
	5	500	64,57 / 15,25 68,85	7,05 / 4,66	3,33 / 4,66	3,35 / 4,17	20,98 / 7,07	99,95 / 100,00	2,9
	11	1000	65,51 / 15,51 69,22	6,75 / 3,83	2,96 / 3,83	3,66 / 4,36	20,98 / 7,07	99,86 / 99,99	2,9
	16	3000	65,32 / 15,25 68,85	6,65 / 4,66	3,61 / 4,66	3,51 / 4,17	21,02 / 7,07	100,11 / 100,00	2,9
	3	5000	65,28 / 15,14 68,87	6,60 / 4,91	3,80 / 4,91	3,15 / 3,75	21,08 / 7,33	99,91 / 100,01	2,9
	4	10000	65,30 / 15,07 68,98	6,56 / 5,04	3,90 / 5,04	3,20 / 3,81	21,04 / 7,09	100,00 / 99,99	2,9

Experimentation, No.	Aging dura- h	Ni	Al	Ti	Cr	W	Sum of ele- ments	Ni	
								$\frac{\text{Ni}}{\Sigma\text{Al, Ti, Cr}}$	
$\gamma'$ - phase iso- lated in an elec- trolyte con- sist- ing of 35 g of citric acid, 5 g of (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> , 15 ml HNO <sub>3</sub> , and 1000 ml of water	15	1000	72,53 / 70,30	8,15 / 17,18	4,19 / 4,98	3,71 / 4,06	11,29 / 3,49	99,87 / 100,01	2,68
	22	3000	72,26 / 70,34	8,00 / 16,95	3,94 / 4,70	3,93 / 4,32	11,84 / 3,68	99,97 / 99,99	2,71
	1	5000	72,87 / 70,95	8,37 / 17,73	3,80 / 4,53	2,77 / 3,04	12,07 / 3,75	99,88 / 100,00	2,80
	2	10000	72,69 / 70,65	8,27 / 17,49	4,14 / 4,93	2,96 / 3,25	11,83 / 3,68	99,94 / 100,00	2,75

**Note.** Above the line—weight percent; below the line—atomic percent.

As can be seen from the data presented, when the aging time of the alloy is varied from 0 to 10,000 h, the composition of the  $\gamma'$ -phase remains, in the main, unchanged. The ratio of Ni to  $\Sigma\text{Al, Ti, Cr}$ , taken in atomic percent, is

approximately 3.

The ratio  $\frac{\text{Ni}}{\Sigma\text{Al, Ti, Cr}}$  in the phase isolated in the electrolyte consisting of 50 ml  $\text{HNO}_3$ , 20 ml  $\text{HClO}_4$  per 1000 ml of water is closer to 3 than in the phase isolated in the electrolyte consisting of 35 g of citric acid, 5 g of  $(\text{NH}_4)_2\text{SO}_4$ , 15 ml  $\text{HNO}_3$ , and 1000 ml of water. This indicates that the first electrolyte is more suitable for isolating the  $\gamma'$ -phase.

According to X-ray analysis\* data, in all anodic powders a  $\gamma'$ -phase was found with lattice parameter  $a = 3.573$  kX, representing a solid solution based on the compound  $\text{Ni}_3\text{Al}$ . Superstructure lines,

\* The X-ray structural study was carried out by A. Ya. Snetkov.

characteristic of the pure compound  $\text{Ni}_3\text{Al}$ , were not found. In all powders isolated from the quenched alloy and from alloys aged up to 10,000 h, weak lines of a phase whose nature has not been established were found on the X-ray patterns. Lines corresponding to the  $\gamma$ -solid solution and to the compound  $\text{Ni}_3\text{Ti}$  were not found.

To study how the elements are distributed between the phases, we carried out a balance of the electrolysis products for an alloy aged for 1000 h, and calculated the content of the elements, in weight percent, in the  $\gamma$ -nickel solid solution and in the  $\gamma'$  phase.

The method we used for electrolytic isolation of excess phases made it possible to establish, in the five-component nickel alloys studied, the presence of a metalloid  $\gamma'$  phase and to examine the effect of prolonged aging time on the composition and structure of this phase.

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*Note: Figure translations are in progress. See original paper for figures.*

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