

A contribution to the Al–Ni–Cr phase diagram

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Abstract

The Al–Ni–Cr phase diagram was specified at 1000 °C and partially at 900 °C. The results concerning the region below 60 at.% Al agreed qualitatively with the literature data. The binary Al–Cr phases μ and γ dissolve up to ~ 1 and ~ 3 at.% Ni, respectively, and Al_3Ni_2 up to ~ 2.5 at.% Cr. Two ternary phases were revealed: hexagonal ζ ($a \approx 1.77$, $c \approx 1.24$ nm) in a wide range between $\text{Al}_{81}\text{Ni}_3\text{Cr}_{16}$, $\text{Al}_{76.5}\text{Ni}_3\text{Cr}_{20.5}$, $\text{Al}_{76.5}\text{Ni}_9\text{Cr}_{14.5}$ and $\text{Al}_{71.5}\text{Ni}_9\text{Cr}_{19.5}$, and high-temperature orthorhombic ε ($a \approx 1.26$, $b \approx 3.48$, $c \approx 2.02$ nm) around $\text{Al}_{76.5}\text{Ni}_{2.0}\text{Cr}_{21.5}$.

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1. Introduction

Although the low-Al part of the Al–Cr–Ni alloy system has been studied since the 1950s, only a few recent publications have been devoted to its high-Al part (see [1] for references). While no ternary phases were reported below 60 at.% Al, investigation of as-cast Al-rich Al–Cr–Ni alloys by scanning and transmission electron microscopy and electron diffraction revealed numerous complicated structures forming at ternary compositions [2]. According to [2], the binary Al–Cr θ -phase (Al_7Cr , also known in the literature as $\text{Al}_{45}\text{Cr}_7$) and η -phases (Al_5Cr , also known in the literature as $\text{Al}_{11}\text{Cr}_2$), whose structures were accepted from [3], exhibit some extensions to ternary compositions. Three ternary compounds designated ρ , ϕ and λ were revealed close to the compositional range of the η -phase. The binary θ -phase and ternary ρ , ϕ and λ phases were reported to exhibit structural variations.

A series of as-cast samples produced in a similar compositional range of Al–Cr–Ni was also studied in [4] by scanning microscopy, powder X-ray diffraction and hardness measurements. The latter study confirmed the existence of three ternary compounds also designated ρ , ϕ and λ and some solubility of Ni in the Al–Cr θ , η and μ (Al_4Cr) phases. However, the phase

fields drawn in [4] were quite different from those in [2]. In addition, wide ternary extensions were drawn in [4] for the Al_3Ni_2 and AlNi phases.

Furthermore, the structure designated ρ_2 in [2] was earlier reported in [5] (so-called κ -phase), but at a composition corresponding to the ϕ -range of Ref. [2]. Also the structure of the Al–Cr η -phase as determined in [3] and accepted in [2] was not confirmed in a more recent study [6]. This shows a need for additional work in order to clarify the constitution of this alloy system.

Due to very different melting temperatures in the high-Al and low-Al regions, the determination of phase equilibria in the whole compositional range of Al–Cr–Ni around 1000 °C would be a very welcome task. For the compositions below 60 at.% Al the 1025 °C isothermal section of the phase diagram has already been determined experimentally (the results are compiled in [7]). Equilibria were established between extended primary solid solutions (Ni) and (Cr) and ternary extensions based on AlNi , AlNi_3 and Al–Cr γ_2 . We guess 1025 °C was selected in order not to be very close to the temperature of the $(\text{AlNi}) + (\text{Ni}) \leftrightarrow (\text{Cr}) + (\text{AlNi}_3)$ reaction, reported first at ~ 1000 °C, but then specified to be at 990 ± 3 °C (see in [7]).

In this contribution, we present the isothermal section of Al–Cr–Ni at 1000 °C and a partial 900 °C section of this phase diagram based on the recent updates of the Al–Cr phase diagram [6,8] and complementary data on the boundary binary systems compiled in [9]. In order to link our results and those in the earlier

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publications on Al–Ni–Cr [7,10,11], several alloys were studied after annealing at 1025 and 1150 °C. Considering the quite extensive information on the low-Al part of the diagram, we examined only a few low-Al samples. The survey of the high-Al range of Al–Ni–Cr below 900 °C will be published elsewhere.

2. Experimental

Alloys of 5–10 g were prepared by inductive melting in a water-cooled copper crucible under an Ar atmosphere. The alloys containing >60 at.% Al were thermally annealed at 1000 °C for 90–120 h and at 900 °C for 110–142 h, those containing <60 at.% Al at 1000 °C for 382–686 h, at 1025 °C for 400 h and at 1150 °C for 49 h. After annealing, the alloys were water quenched. The heat treatments were carried out in alumina crucibles in vacuum or argon-flow furnaces.

The alloys were studied by powder X-ray diffraction (XRD, Cu $K\alpha_1$ radiation was used), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The local phase compositions were determined in the SEM by energy-dispersive X-ray analysis (EDX) on polished unetched cross sections. TEM examinations were carried out on powdered materials dispersed on grids with carbon film. Differential thermal analysis (DTA) was carried out for selected samples. Heating and cooling rates of 10–50 °C/min were used.

3. Results and discussion

The first set of alloys was studied after annealing at 1150 °C, i.e. slightly above the melting temperature of the Al_3Ni_2 phase (δ -phase, incongruently melts at 1133 °C [9]). The results of the examination are shown in Fig. 1a together with the literature data redrawn from Refs. [10] and [11]. At this temperature the Al–Ni–Cr alloy system contains a broad high-Al liquid range which is in equilibrium with the γ_1 solid solution (see below) extended from Al–Cr and the β_{Ni} solid solution based on the congruent AlNi phase. At lower Al concentrations the solid solutions (Cr) and (Ni) based on the corresponding constituent elements and the γ' solid solution based on AlNi_3 were revealed [10,11]. The compositional range between β_{Ni} and (Ni) containing γ' was not studied in the present work and is only shown from the literature data.

According to [8], the Al–Cr γ -range contains a high-temperature cubic γ_1 phase and a low-temperature rhombohedrally distorted γ_2 phase (see Table 1). The temperature

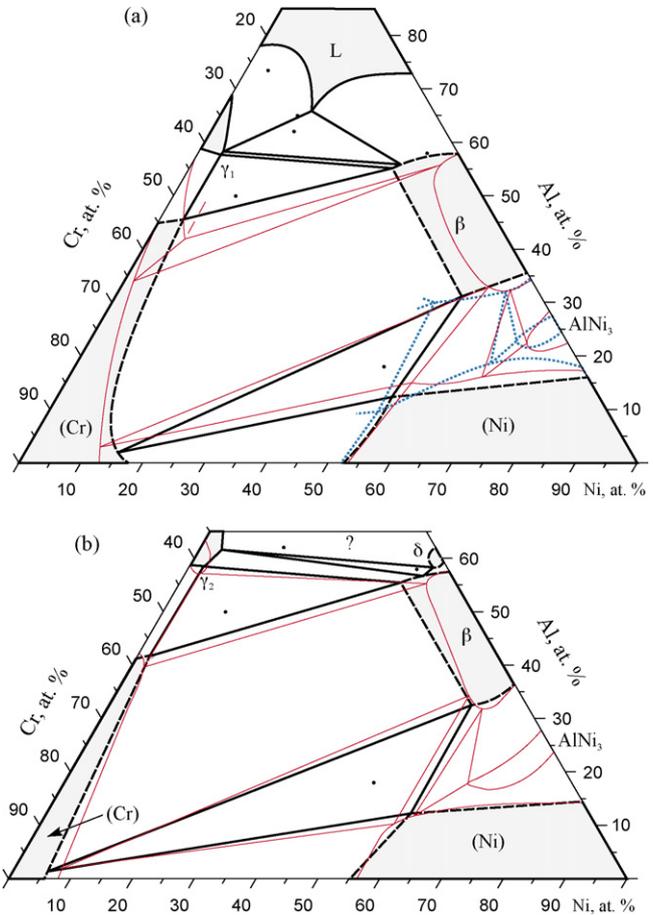


Fig. 1. Isothermal sections of Al–Ni–Cr at: (a) 1150 °C and (b) 1025 °C. The results of the present study are shown by thick lines. The boundaries of the phase ranges (broken lines) are approximated between the corners of the three-phase ranges and the binary compositions of the phases corresponding to the studied temperatures. The compositions of the studied samples are marked by spots, the liquid is designated L. The results for 1150 °C from Ref. [10] are shown in (a) by thin dotted lines (blue) and those from [11] are shown by thin solid lines (red). The results for 1025 °C compiled in [7] are redrawn in (b) by thin lines (red). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

of 1150 °C is above that of the $\gamma_1 \leftrightarrow \gamma_2$ transition in the whole binary compositional range (the transition temperature decreases from 1140 to 1060 °C with increasing Al concentration) [8]. Together with the decrease of the melting temperatures of (Cr) and Al–Cr phases with increasing Ni concentration [1], a decrease of the transition temperatures of the $\gamma_1 \leftrightarrow \gamma_2$ reaction is expected. Therefore, the whole range extended from the Al–Cr γ_1 in Fig. 1a is expected to belong to this structural variant only.

The measured compositions of the phases coexisting in three-phase equilibria visibly deviate from those in both Refs. [10] and [11], which also exhibit visible quantitative differences, but qualitatively all the results are in agreement. The geometry of the widely extended phase boundaries were not specified in our study. The most pronounced difference was revealed in the geometry of our γ_1 range and the corresponding range in [11]. In [11] the low-Al corner of this range “drops” sharply with increasing Ni concentration, so that it almost overlaps with the high-Al limit of the (Cr) range determined in our work. This

Table 1
Crystallographic data of the phases in the Al–Ni–Cr alloy system presented in the isothermal sections of Figs. 1, 3 and 6

Phase	S.G. or symm.	Cell parameters (nm)		
		<i>a</i>	<i>b</i>	<i>c</i>
μ (Al_4Cr)	$P6_3/mmc$	2.00	–	2.46
γ_1^a	$I\bar{4}3m$	0.910	–	–
γ_2^a	$R3m$	1.27	–	0.794
(Cr) ^a	$Im\bar{3}m$	0.288	–	–
δ (Al_3Ni_2)	$P\bar{3}m1$	0.404	–	0.490
β_{Ni} (AlNi)	$Pm\bar{3}m$	0.288	–	–
(AlNi_3) ^a	$Pm\bar{3}m$	0.359	–	–
(Ni) ^a	$Fm\bar{3}m$	0.352	–	–
ζ^a	$P6_3/m$	1.77	–	1.24
ϵ	$Cmcm$	1.26	3.48	2.02

^a Wide compositional range, the lattice parameters are only given for one typical composition.

geometry is rather improbable and may result from a misinterpretation of the measurements in [11]. In our experiments the data for the corresponding compositional triangle were obtained from the three-phase sample of $\text{Al}_{50}\text{Ni}_{10}\text{Cr}_{40}$ (see Fig. 2a) which contained (Cr), while this is excluded according to the phase diagram drawn for 1150°C in [11].

The next experimental temperature of 1025°C is lower than that of the above-mentioned $\gamma_1 \leftrightarrow \gamma_2$ transition in the whole binary compositional range. A decrease of this transition temper-

ature with increasing Ni concentration could in principle result in the stabilization of the high-temperature γ_1 -phase at the experimental temperature. This item was not studied in the present work. Considering a low solubility of Ni in this phase (see Fig. 1a and b) we suggest that the whole γ -range at 1025°C belongs to the γ_2 structural variant. Correspondingly, this is now the (Cr)- β_{Ni} - γ_2 equilibrium instead of the (Cr)- β_{Ni} - γ_1 equilibrium at 1150°C . With the decrease of temperature the concentration limits of (Cr) are shifted towards lower Al and those of β_{Ni} towards the Al–Ni terminal (compare Fig. 1a and b).

SEM/EDX examinations also revealed the δ - β_{Ni} - γ_2 equilibrium and the separation of the β_{Ni} -range from the liquid range. The geometry of the three-phase range (Cr)–(Ni)- β_{Ni} is somewhat different from that at 1150°C . The ranges between (Ni) and β_{Ni} as well as above ~ 60 at.% Al were not examined at 1025°C . Our results concerning the compositions of phases coexisting in the three-phase equilibria are in qualitative agreement with those compiled in [7] (see Fig. 1b), but exhibit some differences in the details. We should mention a quite noticeable scattering of our measurements, which can also be concluded from the data presented in [7]. This could be due to a deviation from equilibria but also small grain size of phases (see Fig. 2b, for example) could result in a decrease in the precision of the EDX measurements.

For the following examinations the annealing temperature was reduced to 1000°C , and the major work was performed in the range above 60 at.% Al. According to the literature data, at 1000°C the solid solution of Ni in bcc Cr extends up to ~ 5 at.% and the solid solution of Cr in fcc Ni extends up to ~ 43 at.%. The solid solution of Al in Ni extends up to ~ 13 at.%. In addition to β_{Ni} and AlNi_3 (γ'), the intermediate δ -phase is solid in the Al–Ni alloy system at 1000°C . The binary compositions of δ range from $\sim \text{Al}_{62.5}\text{Ni}_{37.5}$ to $\text{Al}_{59.5}\text{Ni}_{40.5}$, of β_{Ni} from $\text{Al}_{57}\text{Ni}_{43}$ to $\text{Al}_{37}\text{Ni}_{63}$ and of γ' from $\text{Al}_{26.5}\text{Ni}_{73.5}$ to $\text{Al}_{25.3}\text{Ni}_{74.7}$ [9]. The solid solution of Al in bcc Cr extends up to ~ 42 at.% [9]. At 1000°C apart from low-temperature γ_2 the Al–Cr alloy system also contains the μ -phase [6,8]. In binary compositions the μ -phase extends from $\text{Al}_{79.8}\text{Cr}_{20.2}$ to $\text{Al}_{78.9}\text{Cr}_{21.1}$, while the γ_2 -range widely extends from $\text{Al}_{70.8}\text{Cr}_{29.2}$ to $\text{Al}_{58.1}\text{Cr}_{41.9}$.

The results are presented in the 1000°C isothermal section in Fig. 3a together with those redrawn from [10] for the Ni corner. An enlarged high-Al part of this isothermal section is repeated in Fig. 3b.

The geometry of the γ_2 - β_{Ni} - δ and γ_2 - β_{Ni} -(Cr) three-phase regions remained practically the same as at 1025°C . The ternary extension of the μ -phase periodically forming in Al–Cr at 1040°C [6,8] was found to be ~ 1 at.% Ni, the γ_2 -phase was found to dissolve up to ~ 3 at.% Ni, while the Al–Ni δ -phase extended up to ~ 2.5 at.% Cr. The latter observation is in contrast to the result of [4], where the δ -range was claimed to extend up to 12 at.% Cr. The data in [4] are based on examination of as-cast samples, but according to our examinations even at 1000°C the binary δ -L range is “pressed” to the Al–Ni terminal and at 1025°C the δ -range is still smaller. On the other hand, the β_{Ni} -range is shown by the authors of [4] to extend significantly less than their δ -range and the high-Al limit of the β_{Ni} -range is “rounded”. These are rather erroneous conclusions. According to our results the compositions associated in [4] with the

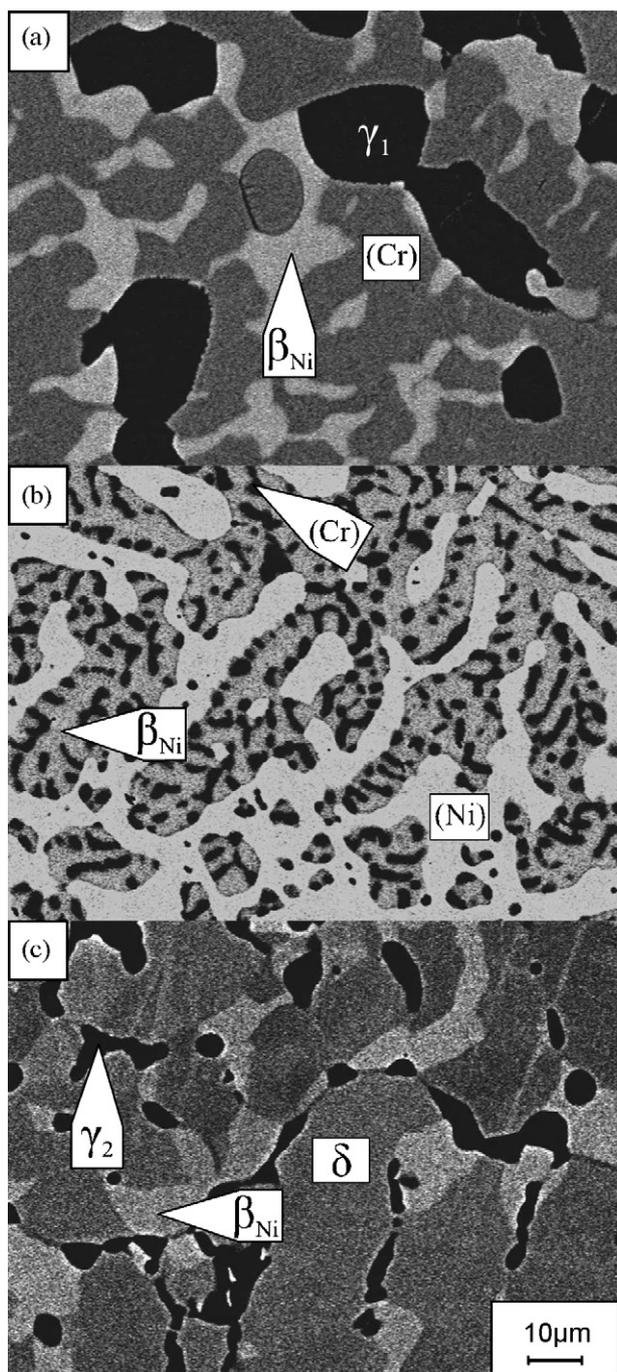


Fig. 2. SEM micrographs (backscattered electron images) of low-Al samples: (a) $\text{Al}_{50}\text{Ni}_{10}\text{Cr}_{40}$ annealed at 1150°C , (b) $\text{Al}_{18}\text{Ni}_{50}\text{Cr}_{32}$ annealed at 1025°C and (c) $\text{Al}_{58}\text{Ni}_{37}\text{Cr}_5$ annealed at 1000°C .

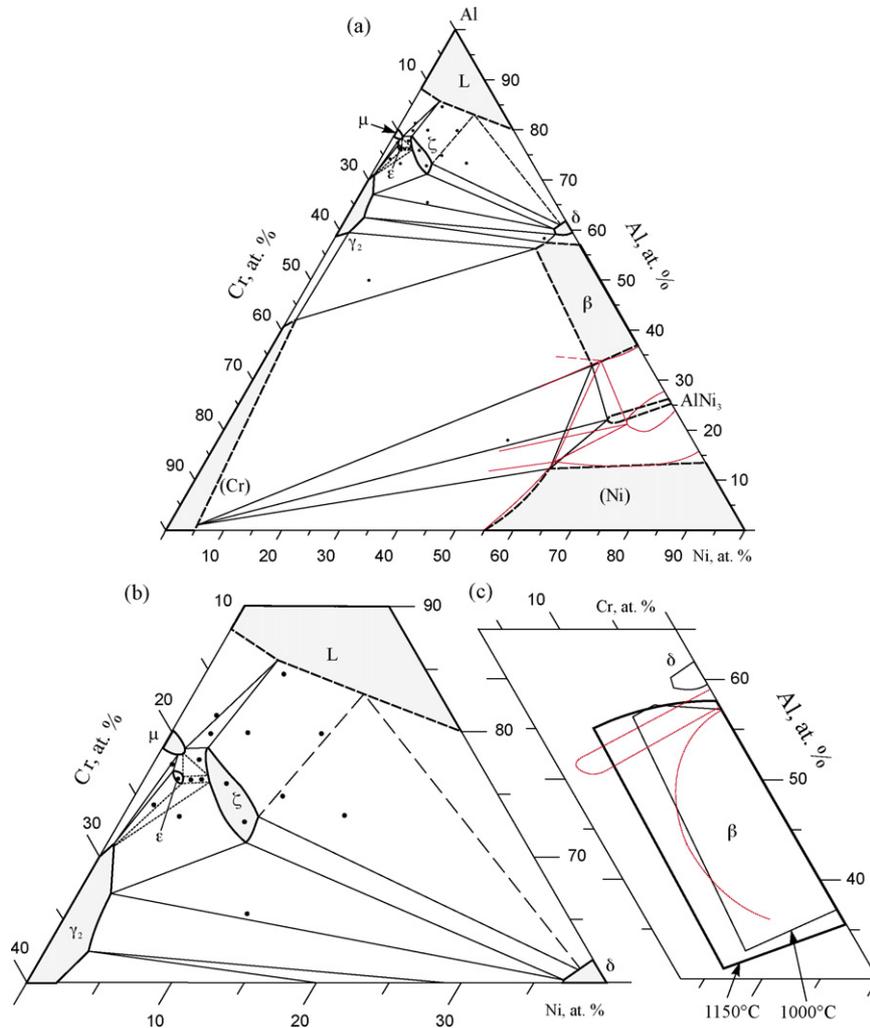


Fig. 3. Isothermal section of Al–Ni–Cr at 1000 °C (a). The results of the present study are shown by thick lines. The boundaries of the phase ranges (broken lines) are approximated between the corners of the three-phase ranges and the binary compositions of the phases corresponding to the studied temperatures. The compositions of the studied samples are marked by spots, the liquid is designated L. The results from Ref. [10] are shown in by thin lines (red). The (Cr)–(Ni)– β_{Ni} and (AlNi₃)–(Ni)– β_{Ni} equilibria are above 1000 °C while (Cr)– β_{Ni} –(AlNi₃) and (Cr)–(Ni)–(AlNi₃) are below 1000 °C. An enlarged part of the isothermal section for high-Al compositions is shown in (b). In (c) the region of the β_{Ni} -phase at 1150 °C (thick solid line, from Fig. 1a) and those of the β_{Ni} -phase and δ -phase at 1000 °C (thin solid lines, blue, from (a)) are compared with the regions of the β_{Ni} -phase and δ -phase redrawn from Ref. [4] (dotted lines, red). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

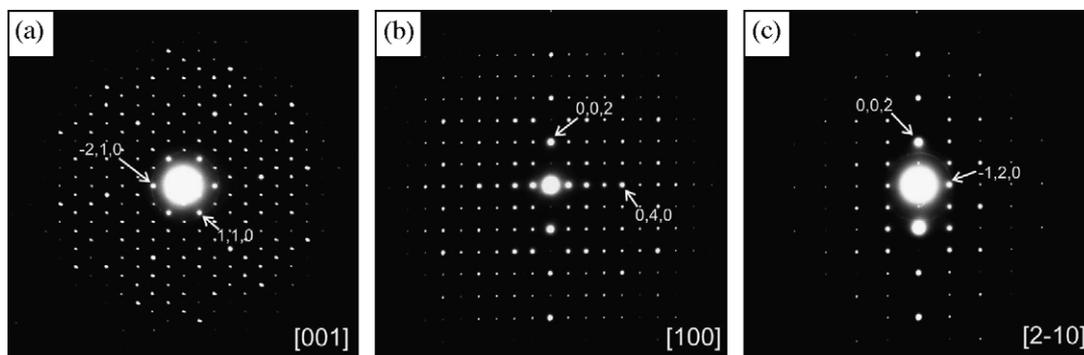
high-Cr end of the δ -range actually belong to the β_{Ni} -range (see Fig. 3c). Despite a narrow compositional gap between the δ and β_{Ni} phases, they are reliably distinguished in metallographic samples (see Fig. 2c, for example).

Two ternary phases were revealed at 1000 °C in the high-Al region of Al–Ni–Cr (see Fig. 3 and Table 1).

Close to the high-Ni limit of the μ -range a ternary phase was revealed in a wide compositional range between about Al_{79.0}Ni_{3.0}Cr_{18.0} and Al_{71.5}Ni_{9.0}Cr_{19.5}. Using electron diffraction its structure was found to be hexagonal ($a \approx 1.77$, $c \approx 1.24$ nm, see Fig. 4) matching the descriptions of the κ -phase of [5] and the ρ_2 -phase of [2]. Such a structure was also observed in the Al–Cu–Cr alloy system (ζ -phase in [12]) in a comparable compositional range. The powder XRD patterns of these phases containing either Cu or Ni were very similar. In order to stress the structural similarity of the phases in both alloy systems we will also use the designation ζ in Al–Ni–Cr. Our DTA examina-

tions the Al–Ni–Cr ζ -phase indicated its formation at ~ 1030 °C. Inside the wide compositional range some variations of its lattice parameters manifested by some shifts of the diffraction lines were revealed.

Close to the μ – γ_2 binary range an additional ternary phase was found to be formed. It exists in a small compositional region around Al_{76.5}Ni_{2.0}Cr_{21.5}. At 900 °C this phase was no longer observed, but the temperature of its decomposition was not detected by DTA. The electron diffraction examinations of an Al_{76.5}Ni_{2.0}Cr_{21.5} sample revealed an orthorhombic structure with $a \approx 1.26$, $b \approx 3.48$, $c \approx 2.02$ nm (see Fig. 5), i.e. the same as associated in [3] with the binary η -phase (Al₁₁Cr₂). Earlier similar electron diffraction patterns were published in [13], where the corresponding structure was also associated with a binary compound. However in contrast to [3], in [13] a slightly lower-Al composition Al₄Cr was claimed for this structure. As was argued in [6], no such structure is formed in the binary Al–Cr alloy sys-

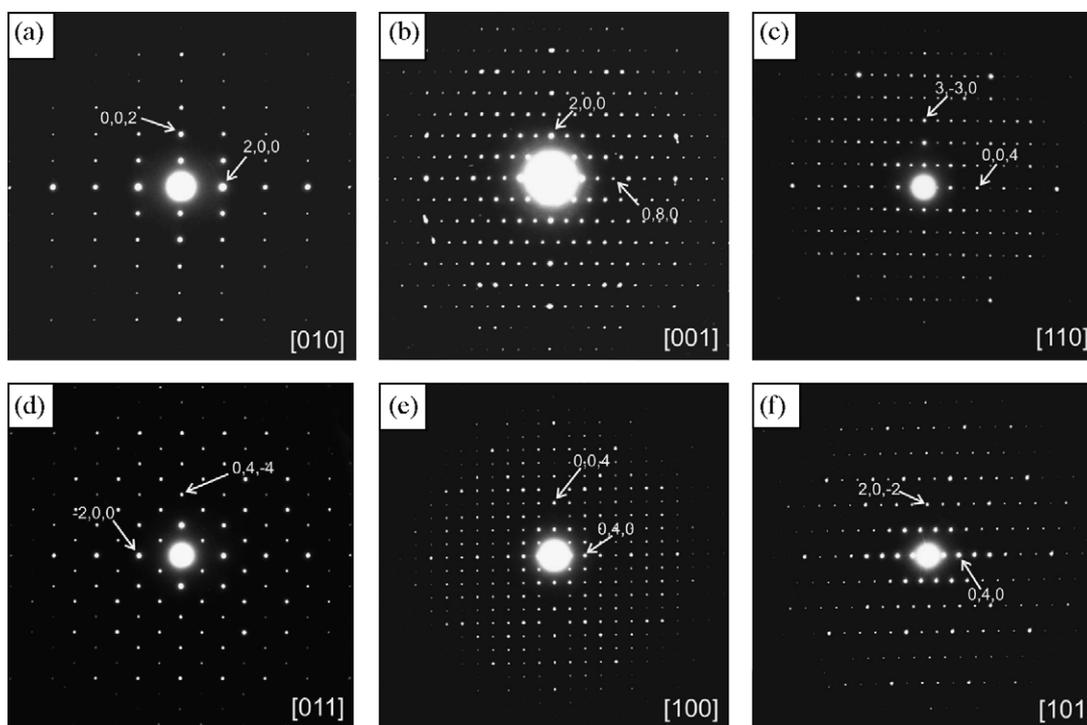
Fig. 4. Electron diffraction patterns of the Al–Ni–Cr ζ -phase.

tem in equilibrium. The observation of this phase, designated in the following ε , in the Al–Ni–Cr alloy system was already mentioned in [6], where its temporary powder XRD standard was used for comparison with those of the binary phases.

The composition determined for our ε -phase matches the low-Al limit of the extended η -range of Ref. [2] fairly well (remember, it was claimed in [2,3] that the η -phase has the same structure as our ε -phase). This is the only compositional correspondence between our results and those in [2]. In contrast to [2], we observed, as already mentioned above, an extension of the Al–Cr μ -region up to ~ 1 at. % Ni and the formation of the ternary ζ -phase at slightly higher-Ni concentrations. At 1000 °C the ζ – μ compositional range separates the range of the ε -phase from the higher-Al part of the phase diagram, where a ternary extension of the Al–Cr η -phase could be expected. The ζ – μ equilibrium was also revealed at 900 °C (see below) and at lower temperatures, at least those temperatures allowed to equilibrate the samples

(to be published elsewhere). It should be stressed that the binary η -phase is formed by a peritectic reaction at ~ 865 °C [6], while the ε -phase decomposes above 900 °C. In addition, examination of other Al–Ni–TM alloy systems (see [14] for references) revealed that the dissolution of Ni in Al–TM phases resulted in only a moderate decrease of their Al concentrations together with a decrease of their melting temperatures, which is also expected in the case of the Al–Cr η -phase. The formation of the ε -phase at almost binary compositions and its close structural relation to the Al–Cr μ -phase [3,13,15] allow us to suggest that it is actually a metastable binary Al–Cr phase stabilized by Ni. The extrapolated binary composition of the ε -phase would be close to the lower-Al limit of the Al–Cr μ -phase.

Two subsequent heat treatments of the $\text{Al}_{18}\text{Ni}_{50}\text{Cr}_{32}$ alloy resulted in the formation of a three-phase microstructure where the grains whose compositions were typical of either (Ni) or (AlNi_3) coexisted with those of (Cr) and β_{Ni} . The second heat

Fig. 5. Electron diffraction patterns of the Al–Ni–Cr ε -phase.

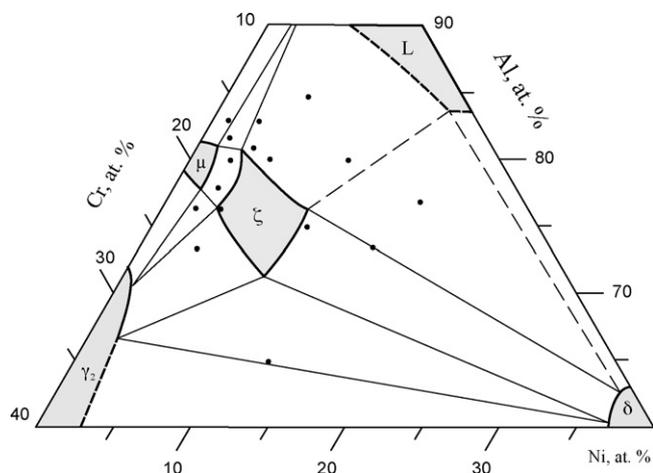


Fig. 6. Partial isothermal section of Al–Ni–Cr at 900 °C. The liquid is designated L. The compositions of the studied samples are shown by spots. Provisional lines are shown by broken lines.

treatment for 304 h was carried out on the same samples previously annealed for 382 h. The phase morphologies observed in SEM in the $\text{Al}_{18}\text{Ni}_{50}\text{Cr}_{32}$ alloy after both heat treatments were very similar. This observation is consistent with the results reported in [10], where the corresponding invariant plane was determined at 1000 °C, rather than with its existence at 990 °C claimed later [7]. Due to small differences in the preset temperatures or temperature gradients in our furnace the actual annealing temperature was once above and once below the transition temperature, which is therefore very close to 1000 °C. Indeed, DTA of the sample exhibited a mixture of (Cr), β_{Ni} and (AlNi₃) revealed a smeared thermal effect close to 1000 °C, but we could not specify the transition temperature with a higher precision. Considering the results obtained at 1025 °C, and in agreement with the literature data, the (Cr)–(Ni)– β_{Ni} and (Ni)– β_{Ni} –(AlNi₃) equilibria are above, while the (Cr)– β_{Ni} –(AlNi₃) and (Cr)–(Ni)–(AlNi₃) equilibria are below 1000 °C. A strong effect at ~1290 °C observed in the same DTA experiment is associated with the $\text{L} \leftrightarrow (\text{Cr}) + \beta_{\text{Ni}} + (\text{Ni})$ eutectic reaction (in [1] the temperature of this reaction is given as 1300 ± 20 °C).

The composition of the (AlNi₃) phase participating at ~1000 °C in the $(\text{AlNi}) + (\text{Ni}) \leftrightarrow (\text{Cr}) + (\text{AlNi}_3)$ reaction, as measured in our experiment, is considerably shifted from the (AlNi₃) range reported in the literature [7,10] (see Fig. 3a). Considering the existence of CrNi₃ isostructural to AlNi₃ [9], one would expect a possibility of forming a continuous solid solution between these binaries, i.e. replacement between Al and Cr in its structure. Since the CrNi₃ structure is only stable below 590 °C

[9], at 1000 °C this could barely be observed, perhaps just as a tendency of the (AlNi₃) compositional range to extend towards CrNi₃. However, the observed shape of the (AlNi₃) compositional range also implies the substitution between Ni and Cr in AlNi₃, which could even be more favorable than between Al and Cr.

The partial isothermal section at 900 °C is shown in Fig. 6. The ϵ -phase no longer exists at this temperature, and the corresponding compositions were found to belong to the ζ – μ – γ_2 ternary range. The range of the ζ -phase becomes wider and extends between about the $\text{Al}_{81}\text{Ni}_3\text{Cr}_{16}$, $\text{Al}_{76.5}\text{Ni}_3\text{Cr}_{20.5}$, $\text{Al}_{76.5}\text{Ni}_9\text{Cr}_{14.5}$ and $\text{Al}_{71.5}\text{Ni}_9\text{Cr}_{19.5}$ compositions. No other ternary phases were revealed at this temperature. Considering the difficulties in achieving equilibrium, no alloys containing less than 60 at.% Al were studied at 900 °C.

Examination of Al-rich alloys at lower temperatures indicated the formation of at least two additional ternary phases. Work on examining them is in progress.

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