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New complex intermetallic in the Al–Rh–Ru alloy system

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ABSTRACT

A ternary orthorhombic phase (*Pbma*, $a = 2.34$, $b = 1.62$ and $c = 2.00$ nm) was revealed around the $Al_{77}Rh_{15}Ru_8$ composition. It is structurally related to the Al–Rh and Al–Pd ε-phases.

1. Introduction

The interest in aluminum alloys with transition metals (TM) and, particularly, with the elements of the platinum group is due to the formation of complex periodic and quasiperiodic intermetallic phases attractive for both basic and applied research. These binary and ternary phases are usually formed in compositional ranges between 60 and 85 at.% Al (see Ref. [1] and references therein). Among them, the so-called ε_l-phases forming in the Al–Pd(or Rh)–(TM) alloy systems are especially interesting. The regular structures belonging to the ε_l family are orthorhombic with essentially the same a and b lattice parameters, while their c lattice parameters are related as $1:(1+\tau):(2+\tau):(3+\tau):(4+\tau)$ etc., where τ is the golden mean. The index of ε_l is the number l of the strong $(0\,0\,l)$ reflection corresponding to the interplanar spacing of about 0.2 nm. In the known ε-phases $l = 6, 16, 22, 28, 34\ldots$ (see Ref. [1] and references therein). In particular, ε_6 and ε_{16} were revealed close to 75 at.% Al in Al–Rh in Ref. [2].[1]

In the present work we report on the revelation of a new Al–Rh–Ru structure, which is related to the ε_l-phases but exhibits a τ-times larger basic structural element.

2. Experimental

Samples were produced by mixing Al–Rh and Al–Ru alloys used to determine the corresponding binary phase diagrams published in Refs. [2,3]. The ternary sample alloys were melted under an Ar atmosphere in an inductive furnace equipped with a water-cooled copper crucible, thermally annealed under an Ar atmosphere at 1100 °C for up to 144 h and subsequently water quenched.

The samples 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 SEM by energy-dispersive X-ray analysis (EDX) on polished unetched cross sections. Transmission electron microscopy and precession electron diffraction experiments were carried out on a 200 kV JEOL FasTEM-2010 electron microscope equipped with an energy-dispersive X-ray spectrometer (NORAN) and spinning star precession unit (Nanomegas). Precession electron diffraction patterns were taken with a nearly parallel beam in nanodiffraction (NBD) mode with a spot size of 15 nm. The degree of precession was in the range of 18.5–46.6 mrad. Images and diffraction patterns were recorded by a Gatan slow-scan digital camera. The TEM study was performed on powdered materials dispersed on Cu grids with an amorphous carbon film.

3. Results and discussion

The new structure was revealed in a small compositional region around the $Al_{77}Rh_{15}Ru_8$ composition, which is somewhat richer in Ru than the ternary extension of the Al–Rh ε-phase(s). Fig. 1 compares the powder XRD pattern of the new ternary phase, designated E, to those of the binary ε-phases.

In order to determine the unit cell geometry of the new E-phase a series of precession electron diffraction patterns of different orientations with large angular separations was recorded. Then three diffraction zones of highest symmetry were selected so, that each pair of patterns had a common side of the rectangular basis as seen in Fig. 2a–c. It was reasonable to assume that the patterns (a–c)

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[1] In Ref. [2] they were designated O_1 and O_2 but in more recent reports were renamed ε_{16} and ε_6, respectively, in order to fit to the nomenclature used for isostructural Al–Pd–(T M) phases.

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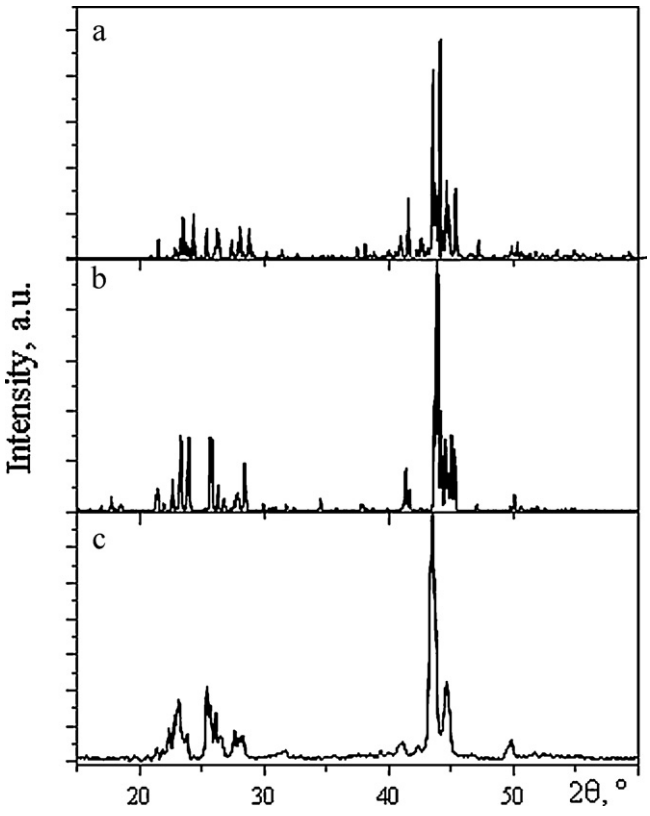

Fig. 1. Powder XRD patterns (Cu $K_{\alpha 1}$ radiation) of the: (a) Al–Rh ε_6-phase, (b) Al–Rh ε_{16}-phase calculated from the data in Ref. [12], and (c) E-phase.

in Fig. 2 could be respectively ascribed to [1 0 0], [0 1 0] and [0 0 1] directions of the orthorhombic crystal lattice, and according to the length of the sides of the rectangular basis the values of the unit cell parameters were estimated as $a = 2.34$, $b = 1.62$ and $c = 2.00$ nm within an accuracy of approximately ±0.004 nm. In terms of this unit cell a successful indexing of all observed diffraction zones was performed thus indicating that the dimensions of the unit cell are correct. The [0 1 0] electron diffraction pattern in Fig. 2b exhibits pseudo-tenfold symmetry, which is typical of the Al–Rh ε-phases (see Ref. [2]). The b lattice parameter of both ε–s and the new structure also corresponds to the periodicity of the decagonal D_4 structure in its specific direction. The decagonal phase of this type (stable or metastable) is observed in several binary and ternary Al–TM alloy systems [1].

Next step in structure determination methodology is evaluation of symmetry of the unit cell. Although convergent beam electron diffraction (CBED) technique is the most suitable approach for this purpose – due to large lattice parameters of the E-phase CBED disks overlapped, which made it impossible to use this approach. Thus, the symmetry of the E-phase was estimated from microdiffraction and selected area electron diffraction patterns using the beam precession technique by Morniroli et al. [4,5]. Since the intensities of the diffracted beams of the precession electron diffraction (PED) patterns are integrated, they are closer to the kinematical intensities [6–8]. Thus, an accurate assessment of the PED patterns provides a possibility of more precisely deriving the extinction conditions and characterizing the space group. Among the possible point groups, corresponding to the orthorhombic system, only *mmm* or 222 can be associated with the 2*mm* symmetry of the zero-order Laue zone (ZOLZ) PED patterns, which is seen in the patterns taken along the [1 0 0], [0 1 0] and [0 0 1] orientations (Fig. 2a–c, respectively). Since the symmetry of the [0 1 1] ZOLZ PED pattern

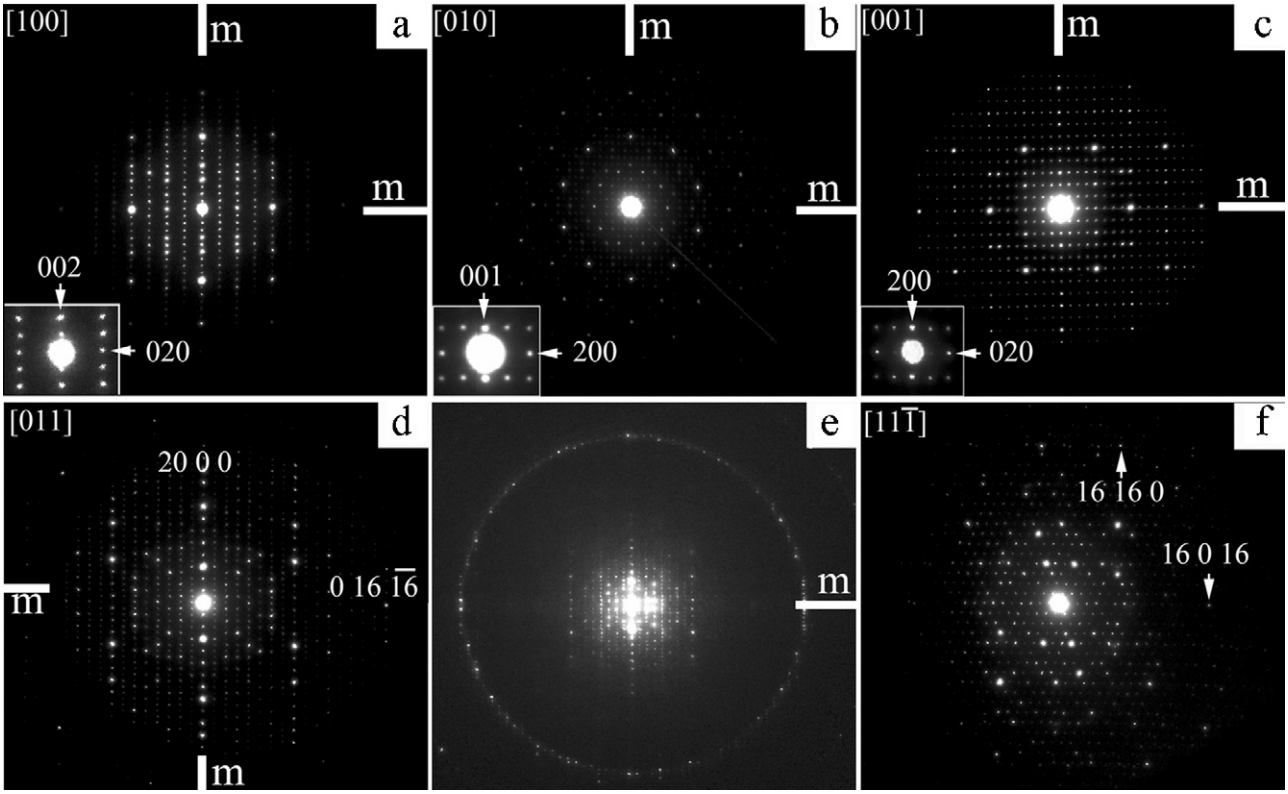

Fig. 2. Electron diffraction patterns taken from the E-phase along: (a) [1 0 0], (b) [0 1 0], (c) [0 0 1] orientations (schematic indexed patterns are shown in the corresponding insets). The mirror planes are labeled by *m*. The precession electron diffraction pattern in (d) and microdiffraction pattern in (e) are taken along the [0 1 1] orientation of the E-phase and the precession electron diffraction in (f) along the [1 1 $\bar{1}$] orientation. The patterns (b), (c), (e) and (f) were obtained with a precession angle of ~2° (34.9 mrad). Reflections with high d_{hkl} value, which appear close to the transmitted beam, have dynamical nature, thus some extra reflections, such as (1 0 1) in Fig. 2f are visible.