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# Intermetallics $La_{11}Ru_2Al_6$ and $Ce_{11}Ru_2Al_6$ with a new structure type

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**The crystal structures of $RE_{11}Ru_2Al_6$ (*RE* = La, Ce) were determined from single-crystal X-ray diffraction at room temperature. The compounds are isostructural and belong to a new structural type: orthorhombic space group *Pbam*, *Z* = 2, *a* = 14.619(2), 14.0799(14) Å, *b* = 15.705(3), 15.4879(16) Å, *c* = 4.4866(7), 4.4685(4) Å, respectively, Pearson symbol *oP*38. An interesting feature of the $Ce_{11}Ru_2Al_6$ structure is the formation of short Ce-Ru distances of 2.4404(8) Å in one of the six crystallographically independent Ce-polyhedra, whereas in the $La_{11}Ru_2Al_6$ structure the La-Ru distances are only slightly shorter than the sum of the covalent radii of La and Ru (2.7033(8) Å).**

**Rare-earth intermetallics / Crystal structure / Crystal chemistry of intermetallics**

## 1. Introduction

In the ternary Ce–Ru–Al compounds with <33 at.% Ce, no chemical bonding peculiarities were found. The compound CeRuAl [1] shows close to covalent Ce-Ru chemical bonding interaction of 2.80 Å. The intermetallic $Ce_5Ru_3Al_2$ [2] was the first aluminide with unusual short Ce-Ru distances (2.5299 Å and 2.5969 Å). In the series of intermetallics with related crystal structures $RE_5Ru_3Al_2$ (*RE* = La, Ce, Pr), neither the La-compound nor the Pr-compound exhibits short *RE*-Ru distances. All the ternary compounds of the Ce–Ru–In system – $Ce_{16}Ru_8In_{37}$ [3], $Ce_2Ru_2In_3$, $Ce_3Ru_2In_2$ [4], $Ce_3Ru_2In_3$ [5], $CeRu_{0.88}In_2$ [6], and $Ce_{16}Ru_{8+x}In_{3-x}$ [7] show short Ce-Ru distances ranging from 2.23 to 2.57 Å. The ternary intermetallics CeRuSn [8], $Ce_2RuZn_4$ [9], $Ce_{23}Ru_7Cd_4$ [10], and $Ce_{23}Ru_7Mg_4$ [11] also show short Ce-Ru distances ranging from 2.40 to 2.70 Å. This chemical bonding is related to the intermediate valence state of some of the Ce atoms in these structures [2,8-11]. Herein, we report on the synthesis and crystal structure of a new compound $Ce_{11}Ru_2Al_6$ with short Ce-Ru distances and its analogue $La_{11}Ru_2Al_6$.

## 2. Experimental

### *2.1. Synthesis*

The synthesis was performed by the reaction of elemental components Ce (99.8%) or La (99.8%), Ru (99.99%), and Al (99.999%) by arc-melting in an argon atmosphere at $0.7 \cdot 10^5$ Pa. On the surface of an ingot of composition Ce60Ru10Al30, single-crystal plates with golden color were found. One of these crystals was used in the X-ray single-crystal diffraction experiment. The melted samples of $RE_{11}Ru_2Al_6$ (*RE* = La, Ce) were placed in evacuated quartz ampoules and annealed at 540°C for 30 days. Afterwards, the ampoules with the samples were quenched in water to room temperature. A needle-like single crystal of $La_{11}Ru_2Al_6$ was found inside an as-cast sample of composition La66Ru17Al17. Crystals of $RE_{11}Ru_2Al_6$ are stable in air for months.

### *2.2. Thermal analysis*

Careful DSC measurements were performed on the two-phase sample of $Ce_{11}Ru_2Al_6$. A piece of 0.025 g mass was heated up to 700°C with a heating rate of 10°C/min in a NETZCH Leading Thermal Analysis STA 449 F1 Jupiter Platinum RT device in a high purity helium atmosphere.

### *2.3. X-ray powder analysis*

Powder diffraction patterns for phase analysis and preliminary determination of unit cell parameters were measured with a STOE STADI P transmission diffractometer, $CuK_{\alpha 1}$-radiation, Ge-monochromator and a linear position-sensitive detector (PSD). The experimental conditions were $10° < 2\theta < 90°$, step scan 0.01° and 10 s counting time per point.

### *2.4. X-ray single-crystal analysis*

The pertinent X-ray single-crystal diffraction data were collected by means of a Nonius Kappa diffractometer with a CCD detector for $Ce_{11}Ru_2Al_6$

**Table 1** Crystal data and structural refinement for $RE_{11}Ru_2Al_6$ (*RE* = La, Ce).

| | | |
|---|---|---|
| Empirical formula | $Ce_{11}Ru_2Al_6$ | $La_{11}Ru_2Al_6$ |
| Molar mass, g/mol | 1905.34 | 1892.03 |
| Unit cell dimensions, Å | 14.0799(14), 15.4879(16), 4.4685(4) | 14.619(2), 15.705(3), 4.4866(7) |
| Space group, *Z* | *Pbam* (No. 55), 2 | *Pbam* (No. 55), 2 |
| *V*, Å$^3$, Calculated density, g/cm$^3$ | 974.44(16), 6.494 | 1030.0(3), 6.100 |
| Crystal size, mm | 0.05 × 0.04 × 0.01 | 0.15 × 0.04 × 0.04 |
| Absorption coefficient, mm$^{-1}$ | 26.837 | 23.893 |
| F(000) | 1608 | 1586 |
| $\theta$ range, ° | 3.91, 34.99 | 1.9, 37.49 |
| Range in *hkl* | ±22, ±24, ±7 | -25 ÷ 18, -22 ÷ 26, ±7 |
| Total no. reflections | 15188 | 12775 |
| Independent reflections ($R_{int}$) | 2340 (0.071) | 2964 (0.050) |
| Reflections with $I>2\sigma(I)$ ($R_\sigma$) | 1571 (0.070) | 2381 (0.044) |
| Data/parameters | 2340/60 | 2964/59 |
| Goodness-of-fit on $F^2$ | 1.045 | 1.021 |
| Final R indices [$I>2\sigma(I)$] R1 | 0.037 | 0.032 |
| *w*R2 | 0.047 | 0.061 |
| R indices (all data) R1 | 0.085 | 0.047 |
| *w*R2 | 0.054 | 0.065 |
| Extinction coefficient | 0.00039(3) | - |
| Largest diff. peak and hole, e/Å$^3$ | 2.220/-3.128 | 2.271/-2.027 |

**Table 2** Atomic coordinates and displacement parameters (Å$^2$) for $RE_{11}Ru_2Al_6$ (*RE* = La, Ce).

| Atom | Multiplicity, Wyckoff letter | *x/a* | *y/b* | *z/c* | $U_{eq}$ |
|---|---|---|---|---|---|
| $La_{11}Ru_2Al_6$ | | | | | |
| La1 | 4*h* | 0.03434(2) | 0.17460(2) | 1/2 | 0.01241(7) |
| La2 | 4*h* | 0.30339(3) | 0.45262(3) | 1/2 | 0.01311(7) |
| La3 | 4*h* | 0.32207(2) | 0.19437(2) | 1/2 | 0.01241(7) |
| La4 | 4*g* | 0.16472(3) | 0.29930(3) | 0 | 0.01252(7) |
| La5 | 4*g* | 0.38819(2) | 0.02488(2) | 0 | 0.01193(7) |
| La6 | 2*a* | 0 | 0 | 0 | 0.01701(11) |
| Ru1 | 4*g* | 0.48094(4) | 0.18174(3) | 0 | 0.01177(9) |
| Al1 | 4*h* | 0.03020(14) | 0.38948(14) | 1/2 | 0.0138(4) |
| Al2 | 4*g* | 0.18716(12) | 0.10005(13) | 0 | 0.0088(3) |
| Al3 | 4*g* | 0.38585(13) | 0.32338(12) | 0 | 0.0090(3) |
| $Ce_{11}Ru_2Al_6$ | | | | | |
| Ce1 | 4*h* | 0.03803(3) | 0.16967(3) | 1/2 | 0.01110(10) |
| Ce2 | 4*h* | 0.29998(3) | 0.45033(3) | 1/2 | 0.01264(10) |
| Ce3 | 4*h* | 0.32231(3) | 0.19470(3) | 1/2 | 0.01081(10) |
| Ce4 | 4*g* | 0.16478(3) | 0.29701(3) | 0 | 0.01281(11) |
| Ce5 | 4*g* | 0.38951(3) | 0.02204(3) | 0 | 0.01136(10) |
| Ce6 | 2*a* | 0 | 0 | 0 | 0.01819(15) |
| Ru1 | 4*g* | 0.49257(5) | 0.18523(4) | 0 | 0.01108(13) |
| Al1 | 4*h* | 0.03437(18) | 0.39152(17) | 1/2 | 0.0136(5) |
| Al2 | 4*g* | 0.18962(16) | 0.09993(15) | 0 | 0.0058(4) |
| Al3 | 4*g* | 0.38436(16) | 0.32340(14) | 0 | 0.0055(4) |

and a Bruker APEX-II CCD diffractometer for $La_{11}Ru_2Al_6$, $MoK_\alpha$, graphite-monochromator. An empirical absorption correction was applied using the program SADABS [12]. The structures of $RE_{11}Ru_2Al_6$ (*RE* = La, Ce) were solved by direct methods (SHELXS97 [13]) and refined by full-matrix least-squares procedures (SHELXL97 [13]) (Table 1).

The atomic coordinates and displacement parameters in the structures of $RE_{11}Ru_2Al_6$ (*RE* = La, Ce) are listed in Table 2. Selected interatomic distances are presented in Table 3.

**Table 3** Selected interatomic distances (Å) for $RE_{11}Ru_2Al_6$ (*RE* = La, Ce).

| Atom1 | Atom2 | Distance | Atom1 | Atom2 | Distance |
|---|---|---|---|---|---|
| $La_{11}Ru_2Al_6$ | | | $Ce_{11}Ru_2Al_6$ | | |
| La1 | 2Al3 | 3.1218(14) | Ce1 | 2Al3 | 3.1120(16) |
| | 2Ru1 | 3.2760(6) | | 2Ru1 | 3.2329(6) |
| | 2Al2 | 3.3756(14) | | 2Al2 | 3.2734(17) |
| | Al1 | 3.375(2) | | Al1 | 3.436(3) |
| | 2La4 | 3.5356(5) | | 2Ce4 | 3.4737(5) |
| | 2La6 | 3.5782(5) | | 2Ce6 | 3.4906(4) |
| | La3 | 3.7234(7) | | | |
| La2 | 2Al2 | 3.2268(15) | Ce2 | 2Al3 | 3.2044(16) |
| | 2Al3 | 3.2566(15) | | 2Al2 | 3.2221(17) |
| | 2La6 | 3.7212(5) | | 2Ce5 | 3.6529(6) |
| | 2La5 | 3.7634(6) | | 2Ce6 | 3.6763(5) |
| | 2La4 | 3.8651(6) | | 2Ce4 | 3.7754(6) |
| La3 | 2Al3 | 3.1633(14) | Ce3 | 2Al3 | 3.1191(16) |
| | 2Al2 | 3.3341(15) | | 2Al2 | 3.2613(17) |
| | Al1 | 3.315(2) | | Al1 | 3.271(3) |
| | 2Ru1 | 3.2351(6) | | 2Ru1 | 3.2803(6) |
| | 2La4 | 3.6109(5) | | 2Ce4 | 3.5245(5) |
| | 2La5 | 3.6128(6) | | 2Ce5 | 3.6108(6) |
| | La1 | 3.7234(7) | | Ce1 | 3.6930(7) |
| La4 | Ru1 | 2.7033(8) | Ce4 | Ru1 | 2.4404(9) |
| | Al2 | 3.146(2) | | Al2 | 3.072(2) |
| | Al3 | 3.255(2) | | Al3 | 3.119(2) |
| | 2Al1 | 3.3024(16) | | 2Al1 | 3.241(2) |
| | 2La1 | 3.5357(5) | | 2Ce1 | 3.4737(5) |
| | 2La3 | 3.6109(5) | | 2Ce3 | 3.5245(5) |
| | La5 | 3.6260(8) | | Ce5 | 3.5680(8) |
| | 2La2 | 3.8650(6) | | 2Ce2 | 3.7754(6) |
| La5 | Ru1 | 2.8119(7) | Ce5 | Ru1 | 2.9144(8) |
| | Al2 | 3.1670(19) | | Al2 | 3.062(2) |
| | 2Al1 | 3.3132(17) | | Ce5 | 3.1854(10) |
| | 2Al1 | 3.3393(16) | | 2Al1 | 3.1979(19) |
| | La5 | 3.3613(9) | | 2Al1 | 3.308(2) |
| | 2La3 | 3.6127(6) | | Ce4 | 3.5680(8) |
| | La4 | 3.6261(8) | | 2Ce3 | 3.6108(6) |
| | 2La2 | 3.7634(6) | | Ru1 | 3.6141(9) |
| | Ru1 | 3.7675(8) | | 2Ce2 | 3.6529(6) |
| La6 | 2Al2 | 3.1552(19) | Ce6 | 2Al2 | 3.086(2) |
| | 2Al3 | 3.237(2) | | 2Al3 | 3.183(2) |
| | 4La1 | 3.5782(5) | | 4Ce1 | 3.4906(4) |
| | 4La2 | 3.7213(5) | | 4Ce2 | 3.6763(5) |
| Ru1 | 2Al1 | 2.6081(12) | Ru1 | Ce4 | 2.4404(8) |
| | Al3 | 2.623(2) | | 2Al1 | 2.5983(14) |
| | La4 | 2.7033(8) | | Al3 | 2.627(2) |
| | La5 | 2.8119(7) | | Ce5 | 2.9144(8) |
| | 2La3 | 3.2351(6) | | 2Ce1 | 3.2329(6) |
| | 2La1 | 3.2760(6) | | 2Ce3 | 3.2803(6) |

**Table 3** Selected interatomic distances (Å) for $RE_{11}Ru_2Al_6$ (*RE* = La, Ce) (continued).

| Atom1 | Atom2 | Distance | Atom1 | Atom2 | Distance |
|---|---|---|---|---|---|
| $La_{11}Ru_2Al_6$ | | | $Ce_{11}Ru_2Al_6$ | | |
| Al1 | 2Ru1 | 2.6081(12) | Al1 | 2Ru1 | 2.5983(14) |
| | 2La4 | 3.3024(16) | | 2Ce5 | 3.1979(19) |
| | 2La5 | 3.3132(17) | | 2Ce4 | 3.241(2) |
| | La3 | 3.315(2) | | Ce3 | 3.271(3) |
| | 2La5 | 3.3393(16) | | 2Ce5 | 3.308(2) |
| | La1 | 3.375(2) | | Ce1 | 3.436(3) |
| Al2 | La4 | 3.146(2) | Al2 | Ce5 | 3.062(2) |
| | La6 | 3.1552(19) | | Ce4 | 3.072(2) |
| | La5 | 3.1670(19) | | Ce6 | 3.086(2) |
| | 2La2 | 3.2268(15) | | 2Ce2 | 3.2221(17) |
| | 2La3 | 3.3340(15) | | 2Ce3 | 3.2613(17) |
| | 2La1 | 3.3755(14) | | 2Ce1 | 3.2734(17) |
| Al3 | Ru1 | 2.623(2) | Al3 | Ru1 | 2.627(2) |
| | 2La1 | 3.1218(14) | | 2Ce1 | 3.1120(16) |
| | 2La3 | 3.1633(14) | | Ce4 | 3.119(2) |
| | La6 | 3.237(2) | | 2Ce3 | 3.1191(16) |
| | La4 | 3.255(2) | | Ce6 | 3.183(2) |
| | 2La2 | 3.2565(15) | | 2Ce2 | 3.2044(16) |

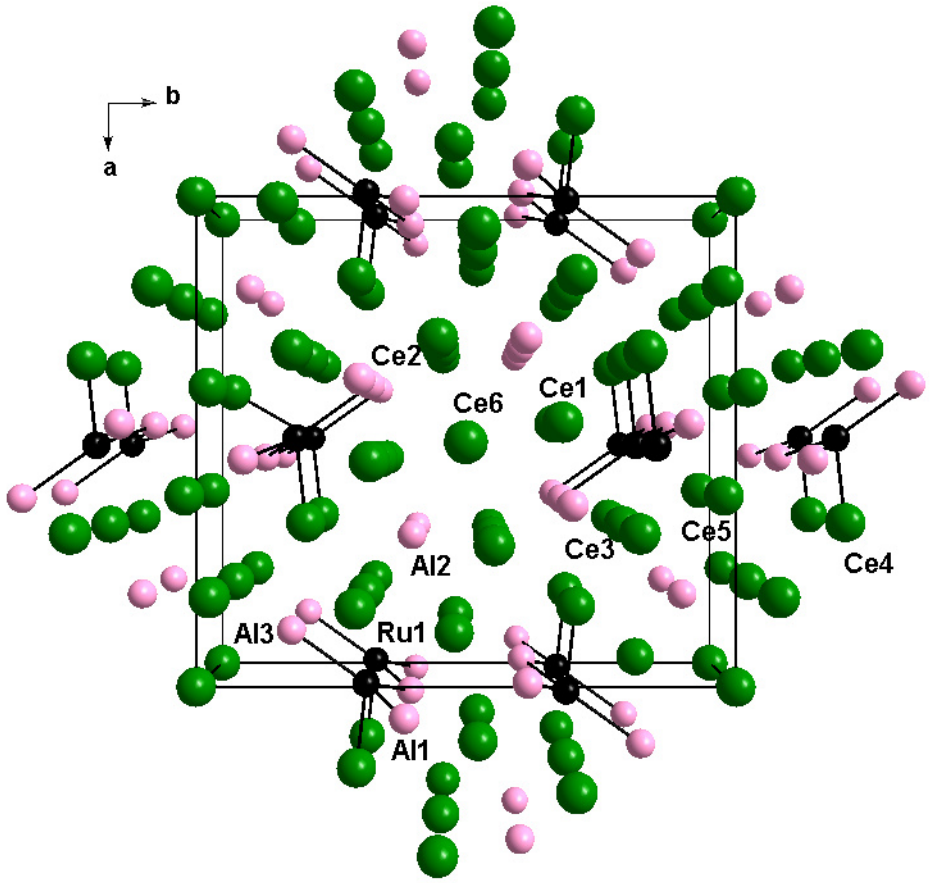



**Fig. 1** View of the structure of $Ce_{11}Ru_2Al_6$ along the [001] direction. The unit cell is outlined.

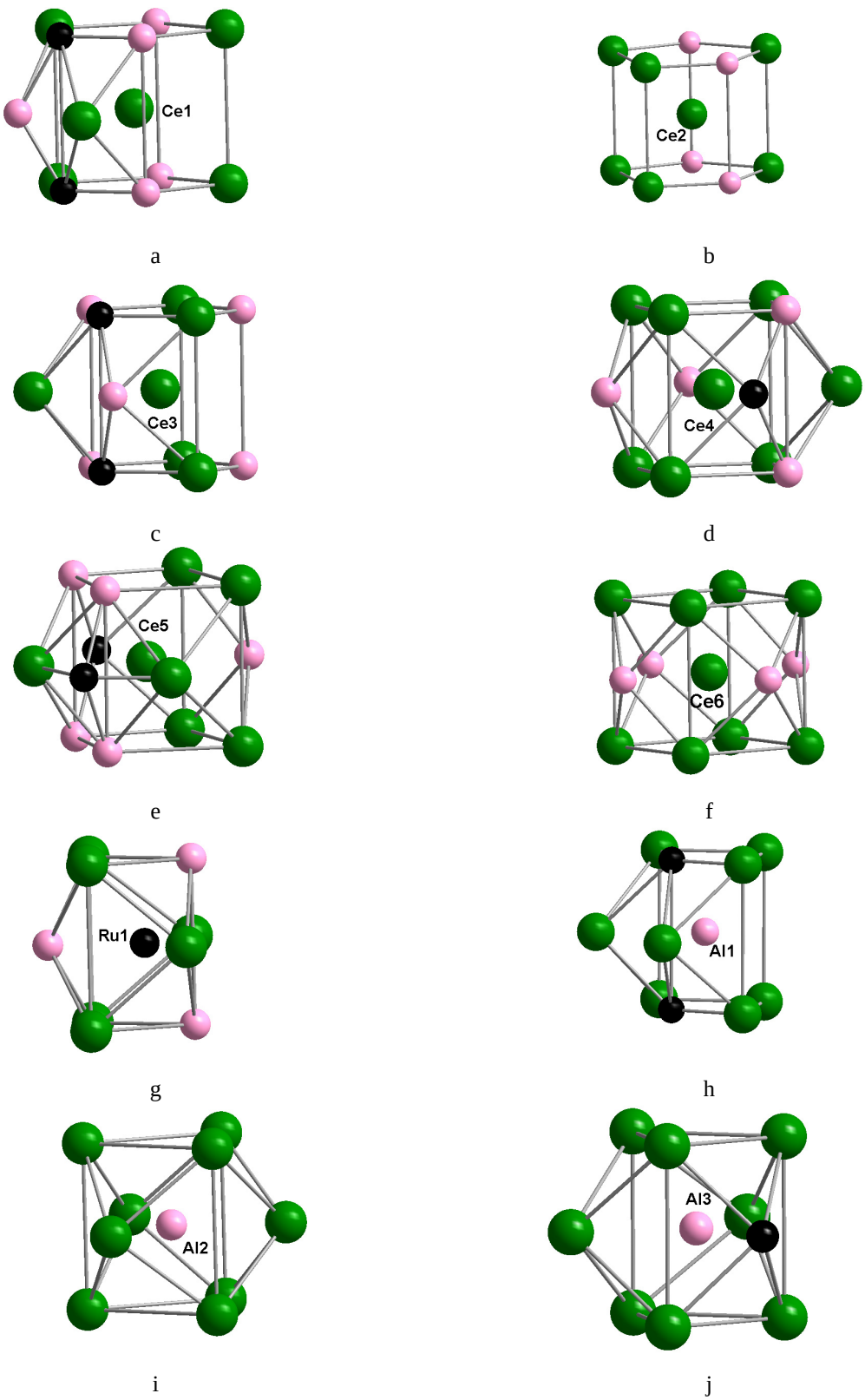
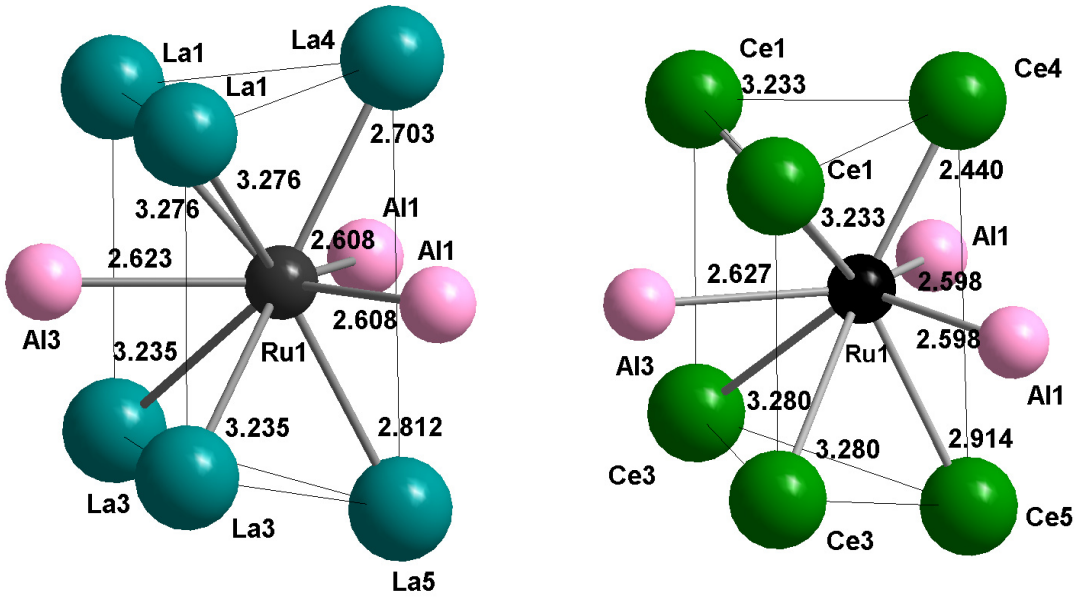



**Fig. 2** The coordination polyhedra of the atoms (a-j) in the structure of $Ce_{11}Ru_2Al_6$.

## 3. Results and discussion

*X-ray powder diffraction and thermal analysis*

We have not obtained $RE_{11}Ru_2Al_6$ in a single-phase state. After annealing at 540°C, the sample of nominal composition Ce57.9Ru10.5Al31.6 consisted of two phases of approximately equal amounts ($Ce_{11}Ru_2Al_6$ and $CeRu_xAl_{2-x}$) [14]. According to the thermal analysis, $Ce_{11}Ru_2Al_6$ melts incongruently at 546°C and shows no polymorphic transition in the




a b

**Fig. 3** Near-neighbor coordination of the Ru1 atoms in a) $La_{11}Ru_2Al_6$; b) $Ce_{11}Ru_2Al_6$.

temperature range from 25 to 546°C. The compound $La_{11}Ru_2Al_6$ was obtained only in the form of a few crystals.

*Crystal structure*

The new intermetallics $RE_{11}Ru_2Al_6$ (*RE* = La, Ce) are isostructural and belong to a new structural type. They crystallize in the orthorhombic space group *Pbam*, $Z = 2$, Pearson symbol *oP*38, cell parameters: $a$ = 14.619(2), 14.0799(14) Å, $b$ = 15.705(3), 15.4879(16) Å, $c$ = 4.4866(7), 4.4685(4) Å, respectively (Table 1). The main differences between the structures of $Ce_{11}Ru_2Al_6$ and $La_{11}Ru_2Al_6$ are connected with peculiarities of Ce-Ru bonding. Anomalously short Ce-Ru distances are caused by the intermediate valence state of some Ce atoms. A view of the $Ce_{11}Ru_2Al_6$ structure along the [001] direction is shown in Fig. 1. The structural motif of $Ce_{11}Ru_2Al_6$ is characterized by infinite chains $[CeRuAl_2]_\infty$ built of Ru1, Al1, Al3 and Ce4 atoms and extending along the [001] direction. The interatomic distances in the chain are shorter than all other contacts in $Ce_{11}Ru_2Al_6$: Ru1 atoms are in contact with two Al1, one Al3, and one Ce4 at the distances 2.5983(14) Å, 2.627(2) Å, and 2.4404(8) Å, respectively. One can suggest that Ru-Al bonding is covalent because the interatomic distances are comparable with the sum of the covalent radii 2.49 Å ($r_{Ru}$ = 1.24 Å, $r_{Al}$ = 1.25 Å) [15]. The Ru1 atom has nine neighboring atoms $[Ce_6Al_3]$ in a tricapped trigonal prism (Fig. 2g). In order to see the influence of the intermediate cerium valence on the local Ru coordination we compared the ruthenium coordination polyhedra in the structures of $Ce_{11}Ru_2Al_6$ and $La_{11}Ru_2Al_6$ (Fig. 3a,b). One can see that in $Ce_{11}Ru_2Al_6$ the Ru1 atom is shifted from the center of the prism towards the Ce4 atom. Then, the Ce4-Ru1 distance becomes shorter, whereas the Ce5-Ru1 distance becomes longer. In the Ru polyhedra of the $La_{11}Ru_2Al_6$ structure, the La4-Ru1 and La5-Ru1 distances are nearly equal. The distances *RE*4-Ru1 and *RE*5-Ru1 in both structures are lower than or equal to the sum of the covalent radii (Table 3).

The aluminum atoms in the structures of $La_{11}Ru_2Al_6$ and $Ce_{11}Ru_2Al_6$ have similar coordination. The Al1 atom is surrounded by a bicapped tetragonal prism of composition $[Ce_8Ru_2]$ (Fig. 2h). The two aluminum atoms Al2 and Al3 have the same coordination of tricapped trigonal prisms: $[Ce_9]$, $[Ce_8Ru]$ (Fig. 2i,j). In contrast to the Al2 polyhedron, in the tricapped trigonal prism around Al3 one of the Ce capping atoms is replaced by a Ru1 atom.

The number of neighbors of the largest size atoms, Ce, varies from 10 to 13 (Fig. 2a-f). The Ce1 and Ce3 atoms have the same coordination of bicapped pentagonal prisms of cerium, aluminum and ruthenium atoms: $[Ce_5Al_5Ru_2]$ and $[Ce_5Al_5Ru_2]$. The Ce2 atom is surrounded by a pentagonal prism of composition $[Ce_6Al_4]$. The two atoms Ce4 and Ce6 have similar coordination of cuboctahedra: $[Ce_7Al_4Ru]$ and $[Ce_8Al_4]$. The Ce5 atom is surrounded by a cuboctahedron with one additional atom $[Ce_6Al_5Ru_2]$.

The atoms Ce1, Ce2, Ce3 and Ce6 are characterized by usual distances from trivalent cerium to surrounding atoms with values of Ce-Al 3.086-3.436 Å, Ce-Ru 3.2329-3.2803 Å, Ce-Ce 3.4737-3.7754 Å (Table 3). By contrast, the Ce4 and Ce5 atoms are surrounded by atoms at anomalous interatomic distances: 2.4404(8) Å and 2.9144(8) Å for Ce4-Ru1 and Ce5-Ru1, respectively, and 3.1854(10) Å for Ce5-Ce5. The short interatomic Ce4-Ru1 distances (less than the sum of the covalent radii

of Ce and Ru) indicate an intermediate valence state of the Ce4 atoms. The Ce5-Ru1 distances of 2.9144(8) Å are close to the sum of the covalent radii of Ce and Ru ($r_{Ce}$ = 1.65 Å, $r_{Ru}$ = 1.24 Å) that can be defined by a valence state slightly higher than three for the Ce5 atom. The short Ce5-Ce5 distances (below the Hill [16] limit 3.40 Å) can lead to overlap of the corresponding 4*f*-orbitals. Similar Ce-Ce short contacts were observed in the structures of α-Ce [17] and $Ce_3Al$-lt [18].

## Conclusion

Ternary intermetallics $RE_{11}Ru_2Al_6$ (*RE* = La, Ce) with a new structure type were for the first time prepared in the *RE*-rich region of the *RE*-Ru-Al systems. $Ce_{11}Ru_2Al_6$ is a new representative of compounds with short Ce-Ru contacts. The significant shortening of the bonding between the Ce and Ru atoms caused by the intermediate valence state of the Ce atoms is typical for the pair of elements Ce and Ru.

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