

## Short Communications

*Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 500 words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible; and proofs will not generally be submitted to authors. Publication will be quicker if the contributions are without illustrations.*

*Acta Cryst.* (1958). **11**, 122

**The tetragonal  $MBe_{12}$  structure of silver, palladium, platinum and gold.** By F. W. VON BATCHELDER and R. F. RAEUCHLE,\* *U. S. Naval Research Laboratory, Washington 25, D. C., U. S. A.*

(Received 17 October 1957)

The structure investigation reported here is a continuation of the work of von Batchelder & Raeuchle (1957). Each compound was obtained by melting, several times, a precious-metal-beryllium mixture of approximately 1:15 atomic ratio in a tungsten arc furnace with a helium atmosphere.

Minute needle-like single crystals were obtained from these melts by electrolytic etch techniques and were examined by the precession method. The lattice constants and space group for  $AgBe_{12}$ ,  $PdBe_{12}$  and  $PtBe_{12}$  were determined from single-crystal precession diagrams taken around the  $a_0$  and  $c_0$  axes. These crystals are tetragonal, and since only the reflection absences characteristics of body centering occurred, the space group is either  $I4/mmm-D_{4h}^7$  or a subgroup. The crystals are isostructural with  $MoBe_{12}$  (Raeuchle & von Batchelder, 1955). The lattice parameters, using  $\lambda(Mo K\alpha) = 0.7107 \text{ \AA}$ , and the calculated densities are reported in Table 1.

Table 1. *Lattice constants for the  $MBe_{12}$  compounds*

Compound	$a_0$ (Å)	$c_0$ (Å)	$c/a$	$D_X$ (g.cm. <sup>-3</sup> )
$AgBe_{12}$	$7.284 \pm 0.005$	$4.246 \pm 0.005$	0.583	3.19
$PdBe_{12}$	$7.271 \pm 0.005$	$4.251 \pm 0.005$	0.585	3.18
$PtBe_{12}$	$7.237 \pm 0.005$	$4.252 \pm 0.005$	0.588	4.53

Each melt was examined spectrochemically and no impurity of any consequence was found. Debye-Scherrer powder patterns were also made from representative portions of each melt. Only the silver-beryllium melt was essentially a single phase which could be indexed as tetragonal  $AgBe_{12}$ . Oesterheld (1916) suggested that an intermetallic compound might exist in the beryllium-silver system at 92.5 atomic% beryllium with the formula  $AgBe_{12}$ .

Powder patterns of the remaining melts show a strong face-centered cubic phase in addition to varying amounts of the tetragonal  $MBe_{12}$  phase. In the diffraction pattern of the palladium-beryllium melt, the strong face-centered cubic phase has the lattice constant  $a_0 = 5.969 \text{ \AA}$  and can be identified with the  $PdBe_5$  phase reported by Misch (1935). The  $PdBe_{12}$  phase was moderately strong.

The powder pattern for the platinum-beryllium melt was indexed as a strong face-centered cubic phase with  $a_0 = 5.978 \text{ \AA}$ , which is probably the compound  $PtBe_5$  (by analogy with  $PdBe_5$ ) and a moderately strong  $PtBe_{12}$  phase. During the single-crystal investigation of this melt, a single crystal of the face-centered cubic phase was obtained and the lattice constant from the precession diagram was found to be  $a_0 = 6.004 \text{ \AA}$ . The discrepancy

between these two values for  $PtBe_5$  is probably due, as suggested by Misch (1935) in the case of  $PdBe_5$ , to the fact that the powder pattern value may have been affected by an excess of beryllium which could lower the lattice constant from the value represented by the true stoichiometric ratio.

The Debye-Scherrer pattern of the gold-beryllium melt was indexed as a very strong face-centered cubic phase with  $a_0 = 6.100 \text{ \AA}$ , which can be identified with  $AuBe_5$  as reported by Misch (1935), and a very weak secondary phase of tetragonal  $MBe_{12}$ . The spectrochemical examination indicated the presence of tungsten. A minute fragment was obtained of essentially a single crystal of the tetragonal phase and examined by the precession camera around  $a_0$  and  $c_0$ . This crystal was found to be isostructural with the tetragonal  $MBe_{12}$  compounds reported, and had lattice constants

$$a_0 = 7.243 \pm 0.005 \text{ \AA}, \quad c_0 = 4.252 \pm 0.005 \text{ \AA}, \\ c/a = 0.587 \quad \text{and} \quad D_X = 4.55 \text{ g.cm.}^{-3}.$$

The composition of this was studied by the electron probe microanalyzer (Birks & Brooks, 1957) and gold plus varying larger amounts of tungsten were found to be present. This would indicate that the fragment examined was not homogeneous in chemical composition. From the X-ray and electron probe evidence, gold and tungsten may jointly form from the melt small percentages of an  $MBe_{12}$ -type of compound. The compound formed could have the possible formula  $Au_xW_yBe_{12}$  (where  $x+y = 1$ ).

A second gold-beryllium melt was made which showed negligible tungsten present. In this the secondary phase was even weaker than in the first melt and no information could be obtained.

We wish to thank Mr D. P. Fickle, of the National Bureau of Standards, for making our melts; Mr S. Cress for spectrographic analysis of our melts; and Messrs L. S. Birks and E. J. Brooks for microanalyzing our small fragment from the Au-Be melt.

## References

- BATCHELDER, F. W. VON & RAEUCHLE, R. F. (1957). *Acta Cryst.* **10**, 648.  
 BIRKS, L. S. & BROOKS, E. J. (1957). *Rev. Sci. Instrum.* To be published.  
 MISCH, L. (1935). *Metallwirtschaft*, **14**, 897.  
 OESTERHELD, G. (1916). *Z. Anorg. Chem.* **97**, 6.  
 RAEUCHLE, R. F. & BATCHELDER, F. W. VON (1955). *Acta Cryst.* **8**, 691.

\* Present address: Olin Mathieson Chemical Corporation, New Haven, Conn., U.S.A.