Technical Note

Preparation and Diffraction Data of Ba-Al Alloys

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ONE of the major uses of barium in metallic form is as a getter material in vacuum tubes. Because of the high chemical reactivity of the metal, Ba-Al alloys are extensively used. Numerous methods for the preparation of Ba-Al alloys have been published, a few of which[1-4] are cited here. Most of these methods were found to be quite elaborate, involving the reduction of BaO, and not too well adapted for the close control of the final composition of small amounts of alloys prepared for laboratory use. A simple laboratory method for the preparation of Ba-Al alloys in small batches starting from pure metals was devised, so that it was possible to control the desired compositions to within 1 pct.

The pertinent features of the alloy system Ba-Al[5] are 1) an intermediate compound $BaAl_4$ with the melting point of 1050°C, and 2) a eutectic between aluminum and $BaAl_4$ at 98 pct Al.

The accompanying sketch shows the experimental arrangement for the preparation of the alloys. Weighed amounts of aluminum and barium were placed in an alumina and a stainless steel crucible, respectively. According to the supplier's specification, the purity of the metals used in the alloys is as follows: a) aluminum rods—99.9 pct Al, and b) barium rods—99.5 pct Ba. The stainless steel crucible, tapered at the bottom and having a 1/16 in. diam hole, rested on top of the alumina crucible. The assembly was placed inside a graphite sleeve which rested on a refractory platform. The platform moved the assembly up and down through the field of a radio frequency coil. A glass bell jar was placed between the crucible assembly and the radio frequency coil to maintain a steady flow of helium around the melt. A small window was cut out on the wall of the alumina crucible to observe the progress of the reaction and to record the temperature with an optical pyrometer.

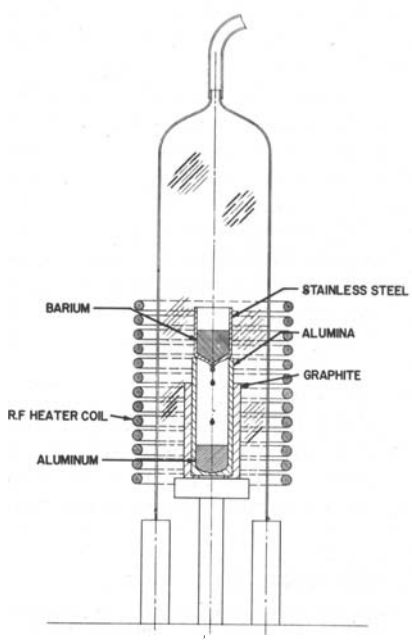

Fig. 1—Experimental arrangement for the preparation of $BaAl_4$.

The platform was first raised high enough to move the barium out of the radio frequency coil field in order to allow only the aluminum to melt. The assembly was then lowered so that the barium began to melt and flow out through the small orifice into the molten aluminum. In order to keep the violence of the exothermic reaction under control, the rate of flow of barium was carefully regulated by raising or lowering the crucible assembly.

All the samples prepared by this technique were examined by a Norelco X-ray diffractometer using Cu$K\alpha$ radiation. The diffraction specimens were prepared by placing the finely powdered samples in flat specimen holders. The Ba-Al alloys prepared with a high barium content were found to consist mainly of $BaAl_4$. The structure of $BaAl_4$ has previously been reported by Alberti and Andress.[6] They found that $BaAl_4$ was body-centered-tetragonal with an $a_0 = b_0 = 4.530$Å and $c_0 = 11.14$Å.

An alloy whose composition was found by chemical analysis to be almost 100 pct $BaAl_4$ was used to determine the relative intensities. The d-spacings were obtained from the same alloy to which a small amount of tungsten had been added as a calibrating material. Accurate values for a_0 and c_0 were calculated according to the method proposed by Taylor and Floyd.[7] The calculated values are: $a_0 = b_0 = 4.566$Å and $c_0 = 11.250$Å. The measured d-values for $BaAl_4$ are shown in Table I along with relative peak intensities above background and hkl indices.

Table I. Powder Diffraction Data of $BaAl_4$

hkl	d, Å	I	hkl	d, Å	I
002	5.60	0.61	206	1.442	0.06
101	4.23	0.74	310	1.435	0.05
110	3.228	0.34	008	1.408	0.13
103	2.902	0.78	224	1.405	0.09
004	2.815	0.66	312	1.398	0.17
112	2.804	1.00	118	1.289	0.05
200	2.282	0.47	217	1.263	0.10
114	2.117	0.11	321	1.258	0.08
202	2.112	0.02	226	1.223	0.02
105	2.023	0.37	109	1.206	0.01
211	2.009	0.20	323	1.198	0.11
066	1.874	0.03	208		
213	1.794	0.40	316	1.144	0.06
204	1.774	0.15	400	1.141	0.05
116	1.624	0.16	0010	1.125	0.01
220	1.615	0.10	402	1.119	0.01
222	1.551	0.03	307	1.103	0.06
107	1.512	0.19	325		
215			411		
301			330	1.076	0.01

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References

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