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The Effect of Water Content on the Formation of Various Aluminum Phosphates

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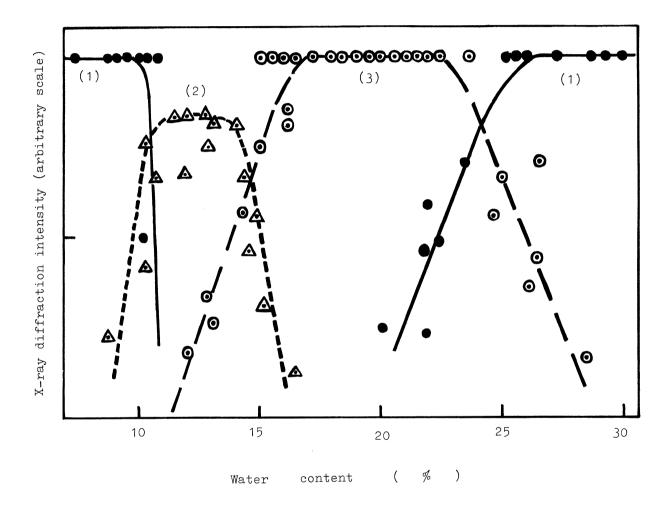
The effect of water content on the formation of various aluminum phosphates from Al, Al_2O_3 or $Al(OH)_3$ and H_3PO_4 was studied. Al₄(P₄O₁₂)₃ is easily formed by the secondary heat treatment when the water content of the primary product is below 10 % or over 25 %, and two unknown substances H and K are formed when the water content is $10 \sim 15$ % and $15 \sim 25$ %, respectively.

It has been reported that various aluminum phosphates were prepared according to the formation conditions, for example the molar ratio R (= Al_2O_3 / P_2O_5) in the material, the heating temperature and the heating time, etc ^{2,3)}. It is also well known from experiment that moisture has a large influence on the formation of these phosphates; however, the relationships between the water content of the material and the formation of various aluminum phosphates are not clear.

In this study, the authors studied the relationships between the formation of various aluminum phosphates and the amount of water in the reactions of d-alumina, γ -alumina, aluminum hydroxide or aluminum metal with phosphoric

acid.

The aluminum raw materials and phosphoric acid were mixed in procelain crucibles so that the molar ratios R in them became 1/3, 1/4, 1/5 and 1/6, and the mixtures were dehydrated by heating over a weak flame with vigorous agitation to obtain white, highly viscous products (the product of this operation is designated as the primary products).



(1): Mixture of the types A and B of $Al_4(P_4O_{12})_3$

(2): The substance H, (3): The substance K

A small amount (0.5 \sim 0.6 g) of each of these products was accurately weighed, and its moisture content was determined by the Karl Fischer method at 300° C, while another portion of it was heated to obtain various aluminum phosphates in a thermostated electric furnace at 300° C for 20 hrs (the product of this operation is designated as the secondary products).

The types A and B of ${\rm Al}_4({\rm P}_4{\rm O}_{12})_3$, 4 $^{\sim}$ 6) an unknown substance H 7) (this substance H, as we tentatively designated, exhibits the characteristic X-ray diffraction peaks at $2\theta = 24.4$, $^{\circ}$, 26.4 and 26.7), and the substance K 2 , 3 , 7) were formed by the secondary heat treatment, and the relative amounts of these aluminum phosphates were determined by the intensities of their characteristic X-ray diffraction peaks, i.e., the type A of ${\rm Al}_4({\rm P}_4{\rm O}_{12})_3$ was determined by the peak at $2\theta = 20.4$, the type B by that at 16.2, the substance H 26.4, and the substance K 11.2.

Figure 1 shows the relationship between the water content of the primary product and the amounts of various aluminum phosphates formed by the secondary heat treatment. The summary of the experimental results is as follows.

- 1) Chiefly $Al_4(P_4O_{12})_3$ is formed by the secondary heat treatment when the water content of the primary product is below 10 %.
- 2) The substance H is easily formed when the water content is about 10 \sim 15 %.
- 3) Chiefly the substance K is formed when the water content is about 15 \sim 25 %.
- 4) However, $\text{Al}_4(\text{P}_4\text{O}_{12})_3$ is formed easily again when the water content is over 25 %.

The same tendency was observed, irrespective of the kinds of the aluminum raw materials used and molar ratios ($1/6 \le R \le 1/3$).

References

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