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DTA STUDIES OF $12CaO \cdot 7Al_2O_3$ POLYMORPHISM

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The polymorphism of $12CaO \cdot 7Al_2O_3$ has been studied by means of a Setaram G–24 thermoanalyser and by X-ray diffraction using a Philips diffractometer. Thermal treatment was carried out at 1.3 Pa as well as in air atmosphere.

The compound $12CaO \cdot 7Al_2O_3$ has been widely studied for a long time. There are many controversies about its polymorphism, structure and other properties in the literature [1–12]. This aluminate forms in self-disintegrating sinters produced in Grzymek's alumina and cement technology [13].

In the present study, differential thermal analysis has been applied for $12CaO \cdot 7Al_2O_3$ polymorphism investigations.

Experimental

The calcium aluminate $12CaO \cdot 7Al_2O_3$ was synthesized from $CaCO_3$ and $Al(OH)_3$ of analytical purity and a fineness below 60 μm ($CaO : Al_2O_3 = 0.94$). The initial mixture was subjected to DTA and X-ray diffraction studies.

The Setaram G–24 thermoanalyser was used at a constant cooling and heating rate of 8 deg/min. The sample was heated at 1793 K during 1 hour and then cooled to 298 K. Some runs were made in vacuum at about 1.3 Pa (samples A in the Figures), and the others in air (samples B in the Figures). X-ray studies were carried out using a Philips diffractometer with CuK_α radiation.

Results and discussion

The two exothermic peaks in Fig. 1, occurring in different temperature ranges, correspond to the crystallization of calcium aluminate from the melt in vacuum (A) and in air (B), respectively. The X-ray diffraction patterns shown in Fig. 2 correspond to the calcium aluminates $5CaO \cdot 3Al_2O_3$ (sample A) and

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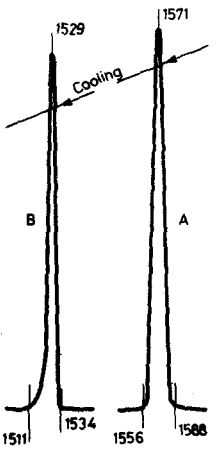

Fig. 1 DTA curves of samples A and B (temp. in K)—cooling

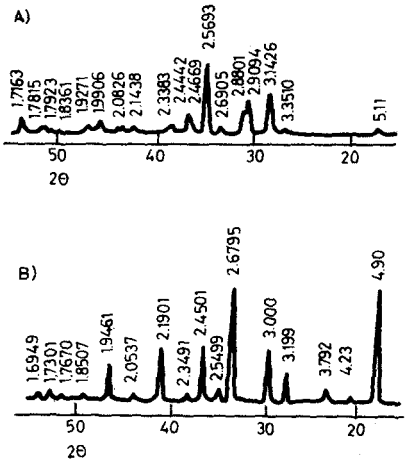

Fig. 2 X-ray diffraction patterns of samples A and B

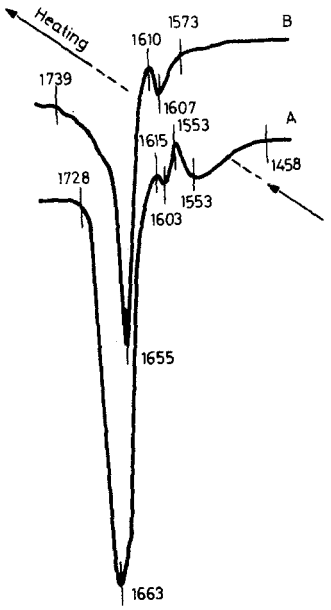

Fig. 3 DTA curves of samples A and B (temperature in K)—heating

$12CaO \cdot 7Al_2O_3$ (sample B) according to the ASTM data. The samples A and B thus obtained were reexamined by DTA in air. As emerges from the DTA curves presented in Fig. 3, the two endothermic peaks can be distinguished in the temperature range 1578–1739 K and the additional third peak for the sample

synthesized in vacuum. The XRD patterns after repeated thermal treatment are identical for the two samples and accord well with the data for $12CaO \cdot 7Al_2O_3$. The authors are therefore of the opinion that the additional peak in the range 1458–1578 K related to the polymorphic transition in $12CaO \cdot 7Al_2O_3$.

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Zusammenfassung — Der Polymorphismus von $12CaO \cdot 7Al_2O_3$ wurde mit Hilfe eines Setaram G-24 Thermoanalysators und durch Röntgenstrukturanalyse mittels eines Philips Diffraktometers untersucht. Die Wärmebehandlung geschah in Luft sowohl bei 1,3 Pa als auch bei Normaldruck.

Резюме — С помощью термоанализатора Сетарам Г-24 и диффрактометра фирмы Филипс изучен полиморфизм $12CaO \cdot 7Al_2O_3$. Термическая обработка была проведена как при 1,3 Па, так и при атмосферном давлении.