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Characterization of Bauxite and its Minerals by Means of Thermoanalytical Methods

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Abstract

Bauxite samples from Boke-Mine, Guinea, West Africa, were investigated by TG-DSC, TG-MS, dilatometry and laser flash analysis. The sample showed mass-loss steps due to the dehydration of aluminum hydroxides and iron hydroxides. Sintering of the samples starts at around 800°C. The thermal diffusivity values were between 1.2 to 0.6 mm²/s for the temperature range RT to 300°C.

Introduction

Mining of bauxite - the main ore for aluminum and alumina production - was in 2009 about 199 Mt [1]. Guinea is the biggest producer of bauxite in Africa and the 5th biggest of the world. These bauxites are so-called lateritic bauxites formed by lateritization of silicate rocks. The samples investigated were from Boke-Mine with a typical composition of 53% Al₂O₃, 16% Fe₂O₃ and minor amounts of SiO₂ and TiO₂ [2].

Experimental

For the TG-DSC measurements of the bauxite samples, a NETZSCH STA 449 F3 Jupiter[®] was employed. The samples were crushed and grinded in a mortar and then measured in Pt crucibles with lids in the temperature range RT to 1500°C. The heating rates were 5 K/min under a flowing air atmosphere with a flow rate of 60 ml/min. The sample weights were approx. 45 mg. For the simultaneous TG-DSC-MS measurements (NETZSCH Aëolos[®] 403 capillary coupling), an argon gas atmosphere with a flow rate of 70 ml/min and a heating rate of 20 K/min were used. The dilatometer measurements were performed with a NETZSCH dilatometer DIL 402 C in the temperature range RT to 1580°C in a dynamic air atmosphere (50 ml/min). The heating rate was 3 K/min. The samples were cut from a solid piece and had a length of 5 mm.

The thermal diffusivity was determined with a NETZSCH laser flash apparatus LFA 447 Nanoflash[®]. The sample was prepared out of a solid bauxite piece. The plan parallel sample had a diameter of 12.7 mm and a thickness of 2 mm. The surface was coated with graphite to avoid internal radiation in the sample.

Results and Discussion

The bauxite samples showed a visual inhomogeneity with red powdery parts and white grains of millimeter size. Figure 1 depicts the TG, DTG and DSC curves of a screening run of a bauxite sample taken from the "red fraction". After a small mass loss of 1.4%, two main TG steps of 9.3 and 8.6% were detected.

These effects came along with the endothermic DSC peaks at 294°C and 501°C with enthalpies of 253 J/g and 267 J/g. At 1023°C, an exothermic DSC peak was observed with an enthalpy of about -51 J/g which is most probably due to the phase transition of γ - to α -alumina. The mass-loss steps are due to dehydration of the aluminum and iron hydroxides proved by the mass spectrometer analysis. In figure 2, the TG and mass spectrometer curves of water and carbon dioxide are plotted. During the TG steps, also small amounts of CO₂ were detected which might be formed during combustion/oxidation of carbonaceous impurities or carbonates. At the phase transition at around 1027°C, a very small amount of CO₂ was also found with no corresponding TG step which proves the high sensitivity of the MS analysis.

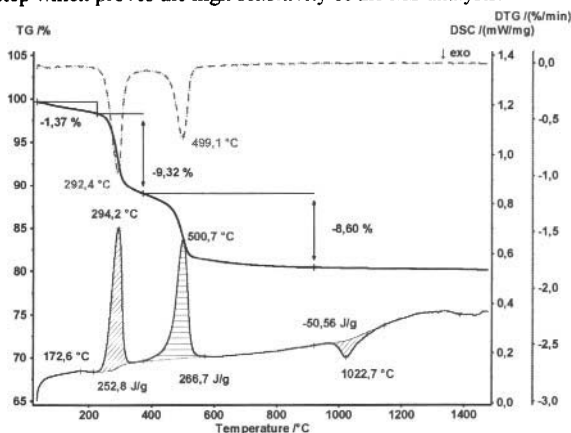


Figure 1. TG, DSC and DTG curves of bauxite "red fraction" at 5 K/min in air atmosphere

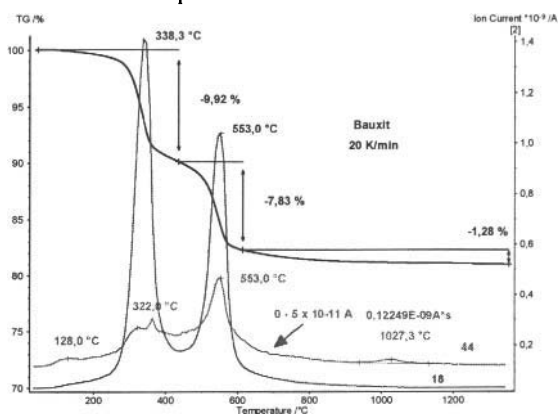


Figure 2. TG and mass spectrometer curves of water (blue curve) and CO₂ (red curve) for bauxite "red fraction" measured at 20 K/min in an argon atmosphere

The TG and DSC results of a more homogeneous sample grinded from white and red parts are shown in figure 3. The heating rate was 5 K/min and the atmosphere was air. It can clearly be seen that the water loss of the 1st TG step is much higher and also the exothermic peak (alumina phase transition) is about a factor 1.6 higher. This means the aluminum content of this sample is reasonably higher than that of the red fraction which is most probably richer in iron. Additionally, endothermic DSC peaks were observed at 1353°C and 1455°C which could be due to phase transitions, partial melting or reduction of iron oxides.

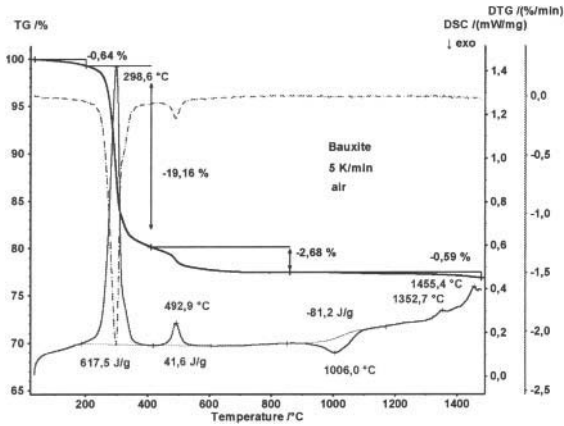


Figure 3. TG, DSC and DTG curves of “homogenized” bauxite measured at 5 K/min in air.

The dilatometer results of a bauxite sample prepared out of a solid piece are shown in figure 4. After expansion from RT to about 250° (linear thermal expansion $\alpha(31, 230) = 1.5 \cdot 10^{-6} \text{ 1/K}$), a shrinkage step of 1.2% was detected at about 309°C (max. shrinkage rate) which corresponds to the 1st TG step (see figure 5). The 2nd mass loss step seems to have little effect on the dilatometer curve of this material. At around 800°C (close to the phase transition), bauxite starts sintering over several steps which is still not finished at 1580°C. The total shrinkage from RT to 1470°C was 25.2%.

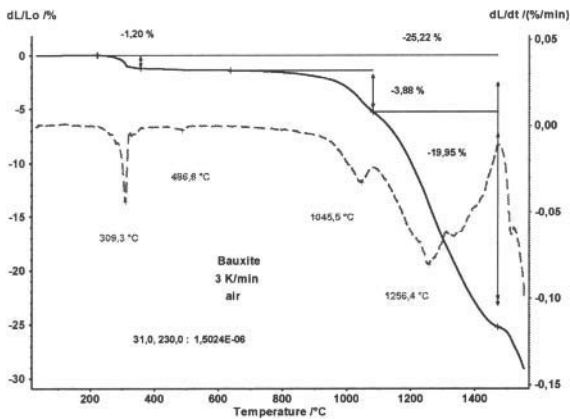


Figure 4. Thermal expansion curve and 1st derivation of bauxite

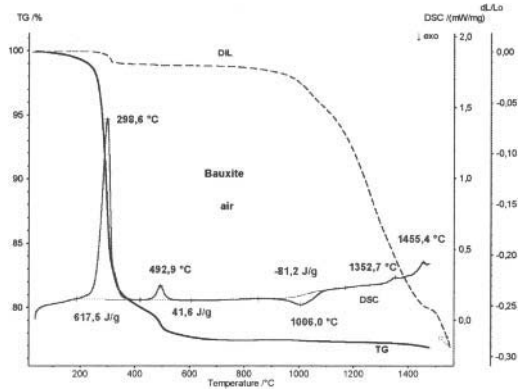


Figure 5. TG, DSC and dilatometer curves of bauxite

The thermal conductivity values of bauxite are shown in figure 6 for the temperature range RT to 300°C. They show the typical decreasing in thermal diffusivity with increasing temperature due to phonon scattering. In comparison to the dense Al_2O_3 standard values, bauxite has a much lower thermal conductivity.

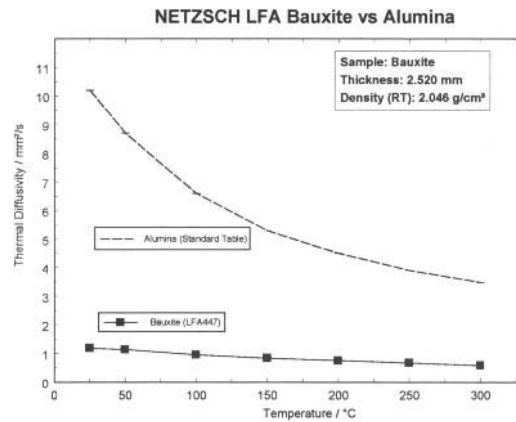


Figure 6. Thermal diffusivity of bauxite compared with alumina

Conclusion

Dehydration of the Boke bauxite happens during two main TG steps at about 300°C and 500°C (DTG maxima). As the samples are inhomogeneous, proper sampling is, of course, very important. The $\gamma - \alpha$ phase transition enthalpy of the Al_2O_3 can give an indication of the alumina content and the homogeneity of the probes. The shrinkage of the bauxite sample from RT to 800°C is only about 1.2%. After 800°C, the sample sintered over several steps by more than 24% at 1500°C. The thermal diffusivity of the bauxite is 1.2 mm^2/s at RT and decreases to 0.6 mm^2/s at 300°C. These values are a factor 5 to 8 smaller than those of the dense alpha-alumina.

References

- [1] USGS 2009 Minerals Yearbook, Bauxite and Alumina,
E. Lee Bradley
- [2] private communication, Aluminium Oxide Stade GmbH