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Aluminium production route through carbosulfidation of alumina utilising H2S

Abstract

Indirect carbothermal reduction of alumina for the production of aluminum utilizes different reducing agents to convert alumina into intermediate aluminum compounds. In the present study, the carbosulfidation route for aluminum production utilizing H2S(g) as the reductant and sulfur source has been investigated, in particular the formation of AhS3 in the first step of the process. The results of the thermodynamic analysis predicted that conversion of Ah03(S) to AhS3(l) significantly increases above 1400°C at 1 atmosphere pressure. Experimental investigations were carried out at temperatures of 1100 to 1500°C using dilute H2S(g) gas in argon. The reaction products were analyzed using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), inductively-coupled plasma absorption emission spectroscopy (ICP-AES) and chemical filtration. The X-ray diffraction results confirmed the presence of AhS3(S). Percentage of conversion from Ah03 to AhS3 was found to be over 80% at 1500°C.

Keywords

route, h2s, production, utilising, aluminium, alumina, carbosulfidation

Disciplines

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Aluminium Production Route through Carbosulfidation of Alumina utilising H_2S

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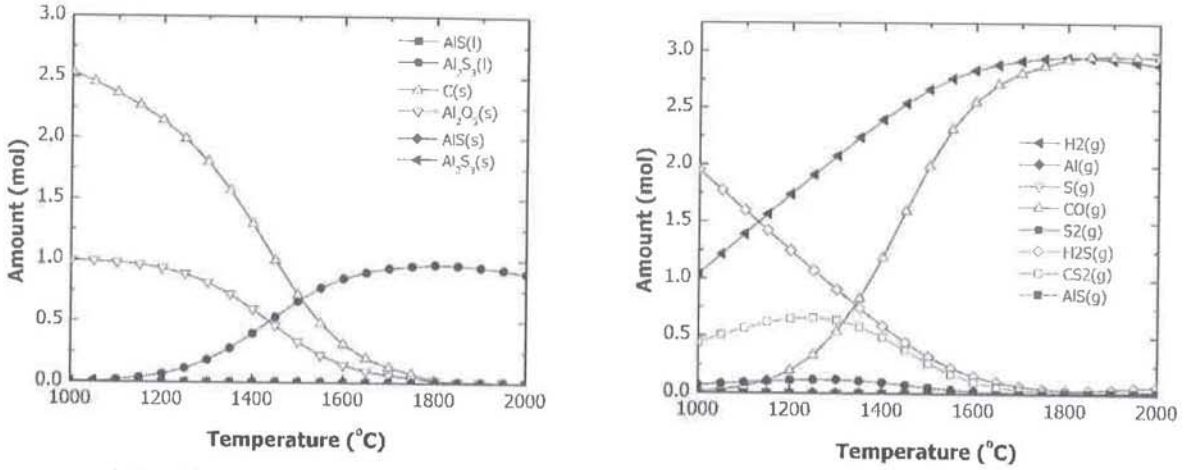
Keywords: Aluminum, carbosulfidation, H_2S

Indirect carbothermal reduction of alumina for the production of aluminum utilizes different reducing agents to convert alumina into intermediate aluminum compounds. In the present study, the carbosulfidation route for aluminum production utilizing $H_2S(g)$ as the reductant and sulfur source has been investigated, in particular the formation of Al_2S_3 in the first step of the process. The results of the thermodynamic analysis predicted that conversion of $Al_2O_3(s)$ to $Al_2S_3(l)$ significantly increases above 1400°C at 1 atmosphere pressure. Experimental investigations were carried out at temperatures of 1100 to 1500°C using dilute $H_2S(g)$ gas in argon. The reaction products were analyzed using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), inductively-coupled plasma absorption emission spectroscopy (ICP-AES) and chemical filtration. The X-ray diffraction results confirmed the presence of $Al_2S_3(s)$. Percentage of conversion from Al_2O_3 to Al_2S_3 was found to be over 80% at 1500°C.

Equilibrium Calculations of Al_2O_3-C-H_2S Reaction Systems

The equilibrium calculations were carried out using FactSage 6.1 thermodynamic package. The equilibrium calculations for Al_2O_3-C-H_2S system were carried at temperatures 1000°C to 2000°C at different pressures. For all equilibrium calculations, 3 moles of C and 3 moles of H_2S were considered for 1 mole of Al_2O_3. Figure 1 shows equilibrium calculation of Al_2O_3+3C+3H_2S for temperature range of 1000 to 2000°C at 1 atm pressure. Figure 1(b) show that significant amounts of gases are produced with majority of $H_2(g)$ and CO(g) at higher temperatures. Al_2S_3 is predicted to be the main intermediate aluminum compound when H_2S is reacted with Al_2O_3 and C at 1000 to 2000°C at 1 atmospheric pressure. Formation of Al_2S_3 is predicted to be very low at 1100 to 1300°C at 1 atm pressure (0.1012 mol Al_2S_3/ mol Al_2O_3) and predicted to increase with increasing temperature to 1800°C. Formation of CO is predicted to be lower at 1100°C (0.035 mol/mol Al_2O_3) and significantly increases with increasing temperature (2.6 mol/mol Al_2O_3 at 1800°C). Along with CO and other gases significant amount of $H_2(g)$ gas is also predicted to form at 1100°C (1.37 mol/ mol Al_2O_3). This content of $H_2(g)$ was predicted to increase to 2.62 mol/mol Al_2O_3 when temperature is at 1800°C.



a) Predicted condensed phases b) Predicted gaseous phases

Figure 1: Predicted equilibrium phases in the Al_2O_3+3C+3H_2S system at T = 1000°C to 2000°C, at 1 atm pressure: a) condensed phases, b) gaseous phases

Experimental results

Experimental investigation on carbosulfidation of Al_2O_3(s) by using C(s) and dilute H_2S(g) (5% H_2S – 95% Ar) at different temperatures (1100 to 1600 °C) and reaction duration were carried out using a horizontal tube resistance-furnace (Nabertherm RHTV 200-600). A schematic diagram of the experimental setup is shown in Figure 2.

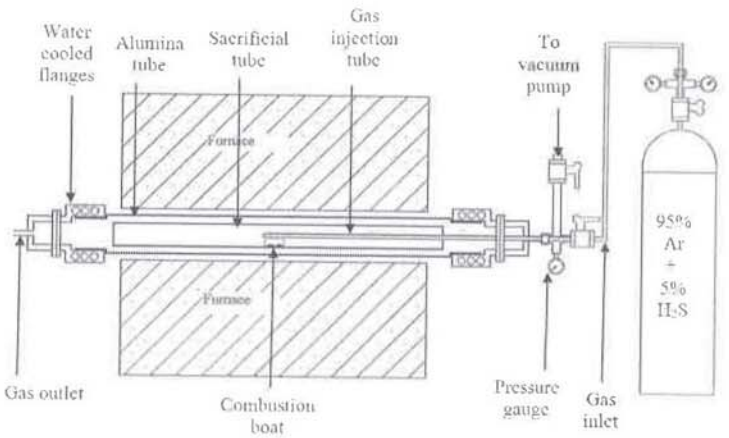


Figure 2: A schematic diagram of the experimental set up using a horizontal tube furnace

Figure 3 shows the comparison of XRD pattern of the samples after experiments at 1400°C for three different times (3, 6 and 9 hours). Al_2O_3 and Al_2S_3 peaks are marked by "1" and "2", respectively. As shown in Figure 3, significant aluminum sulfide (Al_2S_3) was detected after 6 and 9 hours of reaction. This is indicated by the higher and sharper Al_2S_3 peaks at 6 and 9 hours compared to those from at 3 hours. Al_2O_3 peaks are still present, indicated that some Al_2O_3 remains and unreacted in the samples. However, it can also be seen clearly that there is a gradual decrease of the intensity with increasing reaction time.

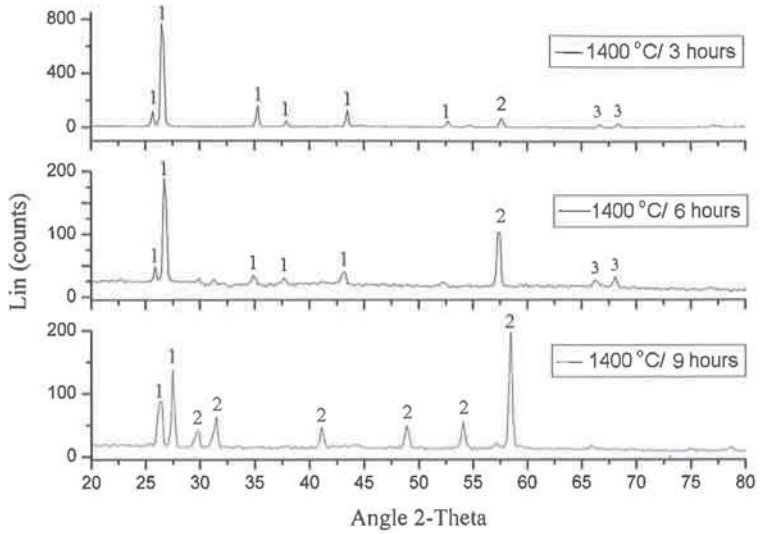


Figure 3: X-ray diffraction pattern of the samples after 3, 6, and 9 hours experiments at 1400 °C. (1 = corundum (Al_2O_3), 2 = aluminum sulfide (Al_2S_3) and 3 = Graphite (C))

The percentage of conversion from Al_2O_3 to Al_2S_3 was determined by chemical dissolution and filtration. As pure Al_2S_3 completely dissolves in hydrochloric acid (HCl), a portion of the experimental samples were dissolved in HCl (36% w/w aqueous solution) and the solution was then filtered out. The amount of mass that dissolves in HCl represents the formed Al_2S_3 while the residues are the unreacted Al_2O_3 and C. From the filtration results, the percent of conversion (η) of Al_2O_3 to Al_2S_3 was calculated using following equation:

$$\eta = \frac{amount\ of\ sample\ dissolved}{amount\ of\ initial\ Al_2O_3} \times 100\%$$

The details of calculated conversion from selected experiments are shown in Table I. The highest conversion was found for experiment at 1500°C and 9 hours duration. The conversion showed an increasing trend with respect to time and temperature.

Table I: The conversion of Al_2O_3 to Al_2S_3 from selected samples at 1400°C and 1500°C

Temperature (°C)	Duration (hours)	Weight of Sample (g)	% of Conversion (η)
1400	6	0.2012	75.4
	9	0.2051	77
1500	6	0.2186	78.9
	9	0.2060	81.6

In summary, the results, from XRD, SEM, EDS, ICP and conversion calculation, indicate that it is possible to form high amount of Al_2S_3 from Al_2O_3 using C and H_2S gas in the range of conditions studied. The results also suggest that the conversion to Al_2S_3 increases with increasing temperature and duration of experiments.