ARCO Metals Company Internal Correspondence 324

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Subject: Conversion of Amorphous PCACH to γ-Alumina

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Following the two previous internal correspondences on the subject dated October 19 and October 31, 1984, the current communication is a summary and wrap-up of our investigations on conversion of amorphous PCACH to γ-Alumina.

Precise determination of alumina phases in PCACH by XRD technique is hindered by non-availability of standards other than α and γ-alumina. Due to this limitation, some PCACH which are designated as γ-alumina may actually be χ (chi) or κ' (kappa prime) alumina. This was evidenced by comparing the scans (Intensity vs. Diffraction angle plots) of sample PCACH with standard scans for α and γ alumina and also by computer phase search.

Based on these observations, presence of α and γ-alumina in PCACH should always be confirmed qualitatively by comparing sample scans with standard ones. Also it was hypothesized that one possible sequence of phase transformation for PCACH could be:

$$\text{Amorphous} \rightarrow \chi \rightarrow \gamma \rightarrow \kappa' \rightarrow \alpha.$$

During the earlier stage of this investigation, it was established that longer calcination time, finer particle size and higher HCl concentration contribute to higher conversion of amorphous PCACH to γ-alumina. It was reported that a calcination time of 60-120 minutes at 600°C was required for high conversion to γ-alumina and the effects of particle size and HCl concentration were minor. Since we are not contemplating any size classification of first stage-calcined material, particle size effect on conversion of amorphous to γ-alumina is not of practical interest. Also, low concentration of HCl (10-15% by volume) is the feasible range for our process. This leaves calcination time as the most significant parameter for the tests to be performed. The other parameter is, of course, temperature of calcination.

In commercial calciners, a retention time of 60 minutes is considered maximum for most applications. For our process, a temperature range of 600-800°C would be considered due to restrictions on calciner material of construction at elevated temperatures. Accordingly, experiments were performed as described by the time-temperature grid shown in Table 1 and the number inside each box indicates the percentage of γ-alumina anlayzed in product PCACH.

Each experiment is started by taking 10 gms of amorphous PCACH in the quartz reactor which is then set up horizontally in the preheated furnace with gas inlet, outlet and thermocouple to monitor bed temperature. Simultaneously gas streams are turned on - HCl (72 ml/min), steam (180 ml/min) and nitrogen (468 ml/min); this corresponds to 10% HCl, 25% steam and 65% nitrogen by volume. The bed temperature rises to the set point and maintained at that temperature for the desired time. At the end of the experiment, HCl and steam are shut off and the reactor is cooled with nitrogen purge and the product PCACH is transferred into a sample bottle for XRD analysis. The results are presented in Table 2. During the recent experiments, the heating time was limited between 9-21 minutes, whereas for previous tests, it is believed to be longer. Higher γ-conversion at 75% for 750C-30M compared to 51% for 750C-45M may be due to longer overall calcination time.

From the data presented in Table 1 and Table 2, it can be concluded that amorphous PCACH can be converted to high concentration γ-alumina (50% or more) at a temperature of 750°C or more and a calcination time of 30-60 minutes would be necessary.

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γ-alumina