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Structural Properties and Surface Characteristics on Aluminum Oxide Powders

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Abstract

\(\alpha\)-Al\(_2\)O\(_3\) is widely used and studied as high temperature structural material, electronic packaging, corrosion resistance ceramics and translucent ceramics. The surface state of \(\alpha\)-Al\(_2\)O\(_3\) powders cannot be regarded as \(\alpha\)-Al\(_2\)O\(_3\) but a hydrated state, and that the nature of this hydrate cannot be considered universal among different \(\alpha\)-Al\(_2\)O\(_3\), even produced by the same production method. One of these reasons is that water can be incorporated in the \(\alpha\)-Al\(_2\)O\(_3\) crystal structure resulting in the formation of aluminum hydroxides such as gibbsite. Although much research has shed light on the Al\(_2\)O\(_3\)-water interface and charging of \(\alpha\)-Al\(_2\)O\(_3\) surfaces, there is still much that we need to better understand \(\alpha\)-Al\(_2\)O\(_3\) structural property and surface characteristics. In this paper, general view of crystal-structural properties, manufacturing methods of high purity \(\alpha\)-Al\(_2\)O\(_3\), and surface characteristics on \(\alpha\)-Al\(_2\)O\(_3\) powders are introduced.

1. Aluminum Oxide

Aluminum oxide (alumina; Al\(_2\)O\(_3\)) has advantages such as its thermal, chemical, and physical properties when compared with several ceramics materials, and is widely used for firebricks, abrasives and integrated circuit (IC) packages. Industrially, more than about 45 million tons of Al\(_2\)O\(_3\) are produced in the world, which are mainly manufactured by the Bayer method using bauxite, and about 40 million tons are consumed for refining aluminum\(^1\) Furthermore about 5 million tons of Al\(_2\)O\(_3\) are produced as chemical grade and used for various purposes. Moreover about 1.5 million ton Al\(_2\)O\(_3\) is used as raw powder in the world. Other Al\(_2\)O\(_3\) is consumed for raw powder of aluminum hydroxide, aluminum sulfate and polyaluminum chloride. The amount of Al\(_2\)O\(_3\) powder used in Japan is about 350,000 tons, what is about 20% of the total quantity produced in the world. In order to produce Al\(_2\)O\(_3\) powder with the quality necessary to be used as ceramic material, various manufacturing methods besides the Bayer method have been developed\(^1, 2\). Depending on the use of Al\(_2\)O\(_3\) in different applications, Al\(_2\)O\(_3\) is classified into different grades. Table 1 shows the different soda level requirements in different applications. Commercial grades of Al\(_2\)O\(_3\) are often divided into: smelter, calcined (milled or unmilled), low soda, reactive, tabular, activated, catalytic, and high purity. These differ in their particle size, morphology, \(\alpha\)-Al\(_2\)O\(_3\) content and impurities.

From crystalline structure difference, there are many forms of Al\(_2\)O\(_3\) (\(\alpha, \chi, \eta, \delta, \kappa, \theta, \gamma, \rho\)). An example of \(\alpha\)-phase of Al\(_2\)O\(_3\) is corundum/sapphire. The other forms are frequently termed transition Al\(_2\)O\(_3\) and arise during the thermal decomposition of aluminum trihydroxides under different conditions. \(\alpha\)-Al\(_2\)O\(_3\) is the most stable form of the compounds formed between aluminum and oxygen, and is the final product from thermal or dehydroxylation treatments of all the hydroxides. Nomenclatures of the aluminum hydroxides are listed in Table 2\(^4\).

The commonly-used Al\(_2\)O\(_3\) is produced through the Bayer process starting from bauxite, which mainly consists of hydrated aluminum. In the Bayer process, crushed bauxite is treated with caustic aluminate solution containing soda. The dissolution reaction is generally carried out under pressure at temperatures ranging from 140 to 280°C. The caustic solution reacts with the aluminum hydroxide so that the impurities can

<table>
<thead>
<tr>
<th>Normal soda</th>
<th>Intermediate soda</th>
<th>Low soda</th>
<th>High purity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.025%</td>
<td>0.15-0.25%</td>
<td>0.03-0.8%</td>
<td>thermally reactive</td>
</tr>
</tbody>
</table>

Table 1: Soda impurity level required for different applications\(^3\)
be separated by sedimentation and filtration, leaving a clear solution. After precipitation of the hydroxide, Al₂O₃ powders can be obtained through heat treatment at their transition temperatures.

2. Production Methods of High Purity α-Al₂O₃ Powder

α-Al₂O₃ powders produced by the Bayer method have maximum purity of 99.6-99.9%, which can be used for manufacturing of refractories, spark plugs, and substrates of integrated circuit. The demand of high purity α-Al₂O₃ is increasing for electronic devices, such as YAG (Yttrium-Aluminum-Garnet) and Titanium Sapphire laser devices. High purity α-Al₂O₃ is indispensable in manufacturing substrate of SOS (Silicon on Sapphire) devices, high pressure sodium lamp, and bioceramics.

High purity α-Al₂O₃ is also used as a high-temperature catalyst and as a support for high-temperature catalysts. The volatile AlCl₃ is oxidized with oxygen or water vapor at 750-900°C, generating a homogenous particle size of 50 nm, consisting of a mixture of α-Al₂O₃ and α-Al₂O₃.

In the conventional chemical vapor deposition (CVD), α-Al₂O₃ with small particle size is produced by a high-temperature reaction at 750-900°C between vaporized AlCl₃ and water vapor.

2AlCl₃ + 3H₂O → Al₂O₃ + 3HCl

The volatile AlCl₃ is oxidized with oxygen or water vapor at 750-900°C, generating a homogenous particle size of fine Al₂O₃ powder. Processing at temperatures of 750-900°C can produce powders with particle size of 50 nm, consisting of a mixture of γ and α-Al₂O₃.

The following equations show the reactions of this method, where R is hydrocarbon radical.

\[\text{Al} + 3\text{ROH} \rightarrow \text{Al}(\text{OR})₃ + \frac{3}{2} \text{H}_2\]

(alkoxide formation)

\[\text{Al}(\text{OR})₃ + 3\text{H}_2\text{O} \rightarrow \text{Al(OH)}₃ + 3\text{ROH}\]

(hydrolysis process)

\[2\text{Al(OH)}₃ \rightarrow \text{Al}_2\text{O}_3 + 3\text{H}_2\text{O}\]

(thermal process)

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\[2\text{Al(OH)}₃ \rightarrow \text{Al}_2\text{O}_3 + 3\text{H}_2\text{O}\]

(thermal process)
such as ruby and sapphire. The equation of heat decomposition is shown below.

\[ 2\text{NH}_4\text{Al} (\text{SO}_4)_2 \cdot 12\text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3 + 2\text{NH}_3 + 4\text{SO}_3 + 25\text{H}_2\]

The ammonium alum is refined mainly by a recrystallization method, weight decreases in 1/9 by the heat decomposition reaction. Therefore, it is necessary to refine to the purity of ammonium alum one figure higher than the target purity of Al$_2$O$_3$. Moreover, it faces the problem about the exclusion processing of the NH$_3$ and SO$_3$ gases which occur at the time of heat decomposition.

1. **Thermal Decomposition of Inorganic Aluminum Salts**

Several inorganic aluminum salts, ammonium aluminum carbonate hydroxide (AACH), NH$_4$AlO(OH) HCO$_3$, for example, can be heated at 230°C to produce Al$_2$O$_3$ through a thermal decomposition shown by the following reaction:

\[ 2\text{NH}_4\text{AlO(OH)HCO}_3 \rightarrow \text{Al}_2\text{O}_3 + 2\text{NH}_3 + 2\text{CO}_2 + 3\text{H}_2\text{O} \]

Production condition of AACH will influence the sintering behavior of α-Al$_2$O$_3$. The process can generate α-Al$_2$O$_3$ powder with purity higher than 99.99% and 0.3-0.4 μm of particle size.

3. **Structure of Aluminum Oxide**

3.1 **Structural Transformations**

Beside α-Al$_2$O$_3$, there are other forms of metastable Al$_2$O$_3$ structures, such as ρ, γ, η, θ, χ and κ-Al$_2$O$_3$. Those kinds of transition Al$_2$O$_3$ can be produced from heat treatment of aluminum hydroxides or aluminum salts.

![Figure 1: Structure transformation of alumina and aluminum hydroxides](image)

The aluminum hydroxides can exist in four well-defined forms: the monohydrate AlOOH, as boehmite (γ-monohydrate) and diaspor (α-monohydrate), and the trihydrate Al(OH)$_3$, as gibbsite (γ-trihydrate) and bayerite (α-trihydrate). Of these, all but bayerite occur naturally in bauxite in profound amounts. At high temperatures, all of the heat treatment paths will terminate in α-Al$_2$O$_3$. Figure 1 shows the paths of transition Al$_2$O$_3$ during the heat treatment processes.

3.2 **Crystal Structure of α-Al$_2$O$_3$**

The crystal structure of α-Al$_2$O$_3$, which is called corundum structure, ideally consists of close packed planes (A and B planes) of large oxygen anions (radius 0.14nm) stacked in the sequence as shown in Figure 2. The aluminum cations (radius 0.053 nm) have valence of +3 and oxygen anions have valence of –2. There can be only two Al$^{3+}$ ions for every three O$^{2-}$ ions to maintain electrical neutrality. Thus, the cations occupy

![Figure 2: (a) Corundum structure in α-Al$_2$O$_3$, (b) top view of the corundum structure, and (c) octahedral structure of α-Al$_2$O$_3$](image)

![Figure 3: Structure of α-Al$_2$O$_3$](image)
only two-thirds of the octahedral sites of the basic array. This placement forms three different types of aluminum cation layers, named a, b, and c.

In Figure 3, the complete stacking sequence of oxygen and aluminum layers will form A-a-B-b-A-c-B-a-A-b-B-c-A···

One period in this sequence, i.e. from c-A to B-c, forms a hexagonal unit cell of $\alpha$-Al$_2$O$_3$.

### 3.3 Crystal Structure of Transition Al$_2$O$_3$

Transition Al$_2$O$_3$ crystallizes in spinel or similar to it with defect lattice. In the spinel structure, the oxygen ions form a face-centered cubic (FCC) lattice and Al$^{3+}$ ions occupy tetrahedral and octahedral interstitial sites, as shown in Figure 4. FCC lattice is also formed by close packed plane, but the stacking sequence is designated as A-B-C-A-B-C···

Since the Al ion favors octahedral coordination under normal circumstances, Saalfeld et al. assumed all octahedral sites to be occupied, and the cation vacancies being confined to the tetrahedral sites. John et al. used solid-state nuclear magnetic resonance (NMR) with magic angle spinning to determine the coordination of Al ions in the transition Al$_2$O$_3$. They found 65 and 75% of the Al ions in the octahedral sites in $\eta$ and $\gamma$-Al$_2$O$_3$, respectively. The results on $\eta$-Al$_2$O$_3$ agree with the X-ray powder diffraction pattern reported by Shirasuka et al. The cation vacancies, therefore, appear to favor octahedral sites in $\eta$-Al$_2$O$_3$ but tetrahedral sites in $\gamma$-Al$_2$O$_3$. The same workers showed that in $\theta$-Al$_2$O$_3$ prepared from both bayerite and boehmite, the Al ions were almost exclusively in octahedral coordination. This is in contrast to Saalfeld’s and Yamaguchi et al. structure analyses of $\theta$-Al$_2$O$_3$ in which both assumed half of the Al ions were occupying the tetrahedral sites. Wefer considered the $\theta$ structure as an intermediate between the cubic close packing of the low-temperature transition Al$_2$O$_3$ and the hexagonally closed-packed corundum.

Ratio of tetrahedral and octahedral sites of Al ions in transition Al$_2$O$_3$ still have contradiction between experimental results observed by several researchers. $\gamma$-Al$_2$O$_3$ has the complex Al-O infrared absorption bands between 350-1100 cm$^{-1}$, which are interpreted under the criteria for the band assignment of the spinels. The positions of the remainder hydroxyl groups are related with the aluminum vacancies.

### 3.4 Crystal Structure of Aluminum Hydroxides

#### 3.4.1 Gibbsite

Pauling first proposed the concept of the gibbsite structure. Double layers of OH ions, with Al ions occupying two-thirds of octahedral interstices within the layers, form the basic structural element. The hydroxyls of adjacent layers are situated, directly opposite to each other, i.e., in a cubic packing. Thus the sequence of OH ions in the direction perpendicular to the planes is A-A-B-B (Figure 5). This superposition of layers and the hexagonal arrangement of Al ions lead to channels through the lattice parallel to the c-axis. Hydrogen bridges OH groups of adjacent double layers. From proton magnetic resonance measurements, Kroon et al. has deduced a model of the spatial distribution of these H-bonds.

#### 3.4.2 Bayerite

Bayerite is rarely found in nature and produced commercially for catalysts or other applications requiring high quality for products. In laboratory, the trihydroxides can be prepared by treatment of aluminum chloride solution with cold ammonium hydroxide, followed by aging at room temperature. According to Fricke et al., bayerite is obtained by hydrolyzing aluminum alcoholates at temperatures below 40°C. Another preparation method is introduced by Torkar et al., who produced extremely pure bayerite electrolytically, using cathode and H$_2$O$_2$ as an electrolyte. Bentor et al. reported the first occurrence of the structure verified by X-ray analysis. The structure of bayerite is similar to gibbsite which is built by basic layers of Al-OH octahedra. The layers are, however, arranged in A-B-A-B-A-B sequence (Figure 4).
Nordstrand et al. published the X-ray diffractogram of a trihydroxide which differed from the diffraction patterns of gibbsite and bayerite. They obtained their trihydroxide by precipitating a gel from aluminum chloride or nitrate solutions with ammonium hydroxide.

There are two kinds of arrangement for the Al-OH octahedra layer, as shown in Figure 6.

**3.4.3 Nordstrandite**

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There are two kinds of arrangement for the Al-OH octahedra layer, as shown in Figure 6.

**3.4.4 Boehmite**

The structure of Boehmite consists of double layers in which oxygen ions are in cubic packing, as shown in Figure 7. These layers are composed of chains formed by double molecules of AlOOH which extend in the direction of the a-axis. The double layers are linked by hydrogen bonds between hydroxyl ions in neighboring planes. Average O-O distance of the hydrogen bridges is 0.27 nm. If the excess water is very high (typically contains > 15 wt% excess water), a, b, c distances in 3 dimensional directions of crystallographic dimension become longer, and produce pseudo-boehmite. X-ray diffraction pattern of pseudo-boehmite is similar to boehmite with a broad peak. Papee et al. postulated that the excess water is not merely adsorbed on crystallite surface, but is located between boehmite-like layers as molecular water. Heating pseudo-boehmite results in the formation of transition Al$_2$O$_3$ in a sequence similar to that associated with bayerite.

**4. Reported Al$_2$O$_3$ Surfaces**

Surface of a solid crystal is regarded as a truncated area of the crystal consisting of coordinately unsaturated site (cus) anions and cations. For this reason, when it is exposed to the atmosphere, all solids become covered with various types of adsorbed species. In the case of metal oxides, the outer layer is usually made up of different adsorbed species. The most abundant component of the surface layer of oxides is water. Besides physisorbed water, absorbed water can be present at the surface in the form of hydrogen bonded or coordinated molecular H$_2$O, and in a dissociated form as surface hydroxyl.

In this regard, infrared spectroscopy has been a commonly used technique to study the surface of Al$_2$O$_3$. By infrared spectroscopy, the O-H stretching fundamental vibrations ($
u_{OH}$) of OH groups located at the surface of oxides can be observed in the high frequency region (wavenumber higher than 2500 cm$^{-1}$). A very broad band centered at around 3300 cm$^{-1}$ has
been assigned to the stretching modes of molecular water hydrogen bonded on an Al₂O₃ surface⁴⁷) and a band at 3500 cm⁻¹ to hydrogen bonded hydroxyl groups. At 870 K, the hydrogen bonds no longer exist and only free hydroxyl groups remain⁴⁶)

The knowledge gained so far about the OH band assignments for transition Al₂O₃ is helpful to understand α-Al₂O₃ powders. The vibrational spectrum from the surface hydroxyl of the transition Al₂O₃ is complex and typical. Table 3 tabulates the bands and assignment proposed for surface hydroxyl species on transition Al₂O₃⁴⁶) so far.

**Table 3:** OH Band assignment proposed for transition aluminas⁴⁶)

<table>
<thead>
<tr>
<th>OH band</th>
<th>Frequency</th>
<th>Peri</th>
<th>Tsyganenko</th>
<th>Knozinger</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3800</td>
<td>A</td>
<td>I</td>
<td>H·O</td>
</tr>
<tr>
<td>2</td>
<td>3775</td>
<td>D</td>
<td>I</td>
<td>H·O</td>
</tr>
<tr>
<td>3</td>
<td>3745</td>
<td>B</td>
<td>II</td>
<td>H·O</td>
</tr>
<tr>
<td>4</td>
<td>3730</td>
<td>E</td>
<td>II</td>
<td>H·O</td>
</tr>
<tr>
<td>5</td>
<td>3710</td>
<td>G</td>
<td>III</td>
<td>bridged</td>
</tr>
<tr>
<td>6</td>
<td>3690</td>
<td>G</td>
<td>III</td>
<td>bridged</td>
</tr>
<tr>
<td>7</td>
<td>3590</td>
<td>H-Bonded</td>
<td>H-bonded</td>
<td>bridged</td>
</tr>
</tbody>
</table>

Actually the spectra of transition Al₂O₃ consist of a mixture of OH bands that closely overlap with each other. Since transition aluminas are commonly used as catalysts or support of catalyst, their surfaces have been attempted to characterize. Several models have been proposed to explain the reactivity of the surface. Several models as summarized by Morterra are described below⁴⁶)

**I: Peri’s model (1965)**

This model advocates that there are five possible free OH configurations on γ-Al₂O₃ surfaces⁴⁸) These are depicted in Figure 8 and their symbol and frequency are shown also in the third column of Table 3. The major limit of Peri’s model consists in the assumption of the (100) crystal face as the only possible termination for aluminas crystallites. This assumption yields an oversimplified picture of the spinel structure, in that only Al³⁺ ions would result present in the uppermost layer and all OH groups in the fully hydrated surface (located on top of equivalent cations) would results to be equivalent. In these conditions, the adoption of random elimination of water from adjoining OH groups is correct and no acidity-basicity concepts associated with different surface OH species needs to be involved. The model is thus valid, in principle, but gives only a partial description of the structurally complex situation of transition aluminas.

![Figure 8](image)

**II: Tsyganenko’s model (1973)**

By considering the most probable terminations of the crystallites and the geometry of the OH groups in these surface termination groups, Tsyganenko et al. came to the conclusion that the number of nearest neighbors has a negligible effect on the frequency of OH species. The determining factor is the number of lattice metal atoms that the OH groups are attached to.

On the basis of the electronic characteristics of oxygen, there can be three types of OH groups, termed OH groups of type I, II, and III, respectively, depending on the coordination number of the OH group⁴⁹) The three types of hydroxyls are shown in Figure 9.

![Figure 9](image)
III: Morterra’s model (1976)

Morterra and co-workers considered the coordination of the Al cations, rather than the coordination of the OH group, as the most important factor in determining the OH frequencies\(^{50)-52}\). According to their approach, that did not consider the actual crystallographic termination of transition \(\text{Al}_2\text{O}_3\) and of the other Al-containing systems, the assignment of the various OH species of \(\text{Al}_2\text{O}_3\) could be made only in very general terms by comparing the OH spectra of different Al oxides. The assignment of OH group is as described in Figure 10.

![Figure 10: Schematic distribution of OH bands in transition aluminas and other Al-containing oxidic systems](image)

IV: Knozinger’s model (1978)

The OH configurations, frequencies and net charges at Al and OH according to this model are given in Table 4.

The net charge is obtained as the sum of the negative charge of the anion and the sum of the strength of the electrostatic bonds (=cation charge divided by coordination number) to the anion from adjacent cations\(^{53}\).

V: The possible role of cation vacancies

The role played by cation vacancies is very difficult to take into account, as there is no way to determine them directly. The possible surface occurrence of cation vacancies is one further parameter, besides OH and cations coordination, that has been considered in the most recent model for OH of aluminas, proposed by Busca et al.\(^{7, 54}\), and Della Gata et al.\(^{55}\).

As a starting point, Busca’s model uses the same general criteria successfully adopted by Knozinger and reported above, but the assignment is also based, on a phenomenological ground, on:

(i) the observation of the OH spectral patterns of several normal spinels (\(\text{MgAl}_2\text{O}_4\), \(\text{ZnAl}_2\text{O}_4\)). Inverse spinels (\(\text{NiAl}_2\text{O}_4\)) and defective spinels (transition phase \(\delta-\text{Al}_2\text{O}_3\));

(ii) the spectral characteristics of metal-hydroxy complexes having non-H-bonded OH groups with different coordination numbers.

The conclusions reached, that are schematically reported also in the last column of Table 3, reassign the various OH species of aluminas as reported in Figure 11.

![Figure 11: Possible OH structures, and \(\nu\text{OH}\) frequencies, at the surface of defective spinel transition aluminas (Symbol: | stands for a cation vacancy)](image)

VI: Morterra’s defect model (1994)

Finally, Morterra et al. attributed the 3775 cm\(^{-1}\) OH band to hydroxyl group coordinated on tetrahedral Al ion (\(\text{Al}^{IV}-\text{OH}\) groups) present in portions of surface belonging to crystallographically defective configurations (i.e. stepped terminations) which frequently occur in porous system of high surface area and poor crystallinity\(^{56}\).

Enormous amount of infrared spectroscopic works have been devoted to understand the surface behavior of \(\text{Al}_2\text{O}_3\), since Peri proposed a model of \(\text{Al}_2\text{O}_3\) about thirty years ago. Most of the works have been related to transition \(\text{Al}_2\text{O}_3\). In spite of some controversial aspects and some remaining uncertainties of the results, the overall picture obtained for the surface properties of the transition \(\text{Al}_2\text{O}_3\) may be useful for understanding the surface behavior of \(\alpha-\text{Al}_2\text{O}_3\) in this study.

5. Concluding Remarks

Crystal structural properties, manufacturing methods of high purity \(\text{Al}_2\text{O}_3\), and surface characteristics on \(\text{Al}_2\text{O}_3\) oxide powders are described.

The surface chemical characteristics of materials play an important role in many technological processes and applications. Differences in surface conditions of the powders can influence their physical properties such as zeta potential, powder agglomeration and sintering.
behavior. Ceramics, unlike metals, are mainly produced by sintering, which implies that the surface structure of the raw powder is a controlling factor to determine the final characteristics of sintered ceramics. Control of these behaviors is useful in developing cost effective ceramic powder processing schemes to produce reliable high-performance ceramics with minimal reject rates.

6. Acknowledgement

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7. References


— 30 —


