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Studies on the Formation of Ferrite by the Wet Method

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In recent years, in the field of ceramics attention has been payed to the use of fine raw materials with the object of getting the sintered piece by heating at comparatively low temperature. By using fine materials, it has been also looked upon producing high density or low porosity ceramics.

Although it has been already known that fine ferrite particle is able to be synthesized by the wet method especially the hydrothermal method, lately the time is getting ripe for using fine ferrite particle practically.

For practical use, it is proper to grasp their properties in detail and in connection with them it is important to solve the mechanism of ferrite formation by the wet method. However, as a matter of fact, it has been insufficient and inconvenient to study it fundamentally. Therefore the study was begun to make clear the mechanism.

In the case of the hydrothermal method, it is impossible to produce ferrite continuously for reasons of the equipment. So this method is inconvenient to use practically. In order to improve this point, the method to heat the raw materials under water vapor was designed. On account of this reaction, fundamental experiment was made and the mechanism of ferrite formation by this method was discussed. Moreover, necessary things for application was followed after.

In this study, the coprecipitates, the wet mixture and the dry mixture were used as starting materials. These are reacted by the hydrothermal method and the heating under water vapor method. Nickel, magnesium and zinc ferrite were synthesized. Samples after these treatment were put to magnetobalance analysis, thermobalance analysis, X-ray analysis and observation by an electron microscope.

As the result of this experiment, it becomes clear that phenomena happened in hydrothermal reaction is similar to that happened in heating under water vapor but there is a little difference between them.

In general, magnesium ferrite formation was said to be impossible by the hydrothermal method. So the reason why magnesium ferrite was difficult to be formed was discussed and it was successful to synthesize it by the hydrothermal method. The condition is that magnesium ferrite coprecipitate has to be washed sufficiently before reaction and pH of the suspension is kept around 10.5 during reaction. This pH of the suspension is a little low compared with that for ferrite coprecipitate of other composition.

It was also difficult to synthesize magnesium ferrite by heating under water vapor.

But after much trial experiment, magnesium ferrite was easy to be formed by adding hydrogen chloride gas or substances which generates hydrogen chloride during reaction.

Zinc ferrite was found to be formed by the hydrothermal method from the mixture of geothite and zinc oxide. In the case of using the coprecipitate as the raw materials, it is impossible to observe the shape of ferrite particle. But on the contrary, it is easy to observe the shape by using the dry mixture. By observing the ferrite particle shape by an electron microscope, grain growth of zinc ferrite was recognized. The factor for controled grain growth was discussed.

With the object of guessing the mechanism of ferrite formation by the wet method, synthesis of ferrite was pursued from the standpoint of reaction velocity. As the result, the quantity of ferrite formation is proportional to logarithum of the reaction time both in hydrothermal reaction and heating under water vapor.

On the basis of the result of these studies and references, the outline of the mechanism of ferrite formation by the wet method is discussed collectively and guessed roughly which has been scarecely made clear up to date.

The coprecipitate and the wet mixture have the imperfect lattice structure which is similar to spinel structure. But homogeneity of constituent ions depends on sort and concentration of metalic salt aqueous solution and alkali aqueous solution. It is also based on the way of precipitation.

In the case of the coprecipitate and the wet mixture ferrite is formed by rearrangement with short-range movement of constituent ions in the imperfect lattice.

On the other hand, in the case of dry mixture, ferrite is formed by chemical bonding of the complexes with dehydration which is supplied from the raw materials into a medium.

In hydrothermal reaction, an amount of ferrite formed and its grain size are under the control of treatment temperature, time, pH and ionic atmosphere.

In heating under water vapor, an amount of ferrite formed depends on reaction temperature, time, water vapor pressure and additives.

The shape of ferrite formed by the wet method is fine sphere or cubic.

Magnetically it is superparamagnetic because of it's small grain size. Usually it is from 100 Å to 500 Å in diameter.

Applications of these fine grain ferrite is described. Fine grain materials are thought to be active. Therefore, it is possible to produce fine grain dense ferrite at comparatively low temperature.

Problems left behind are as follows: First of all dehydration mechanisms at reactions have to be studied much more in detail. Reaction condition should be tried to be found out to form ferrite with nonstoichiometric composition. For better application of the properties of this fine ferrite, a special production method for sintered pieces should be established.