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More Uniform Ferrite Powders through Precise Ceramic Processing

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Abstract. The ferrite industries are expected to grow continuously and an estimated covering the market development of forthcoming years, in terms of produced annual tonnage, will be presented. The creation of uniform sintered structures, within the lower micron meter range, essentially free of duplex structures, is highly desirable for the production of a large proportion of soft and hard ferrite products. Several wet chemical processes have been proposed and are presently being carried out on a commercial scale for the production of ferrite powders. For the production of precisely controlled uncollapsed ferrite powders, exhibiting uniform primary particle sizes within the submicron meter range, should for cost savings, environmental and constant quality control purposes, be carried out at selected and integrated steel mills using short thermal sintering techniques. The advantage of an independent management - away from the core business steel - would be of mutual benefit to the ferrite and steel industries.

1. Estimated World Ferrite Production

An estimate covering the world-wide ferrite production for the period of 1990 up to 2005 is shown in Table 1. The annual growth rates for indicated ferrite applications are expected to vary between 5 and 15 percent. Total market value of ceramic and plastic bonded ferrites for 1995 may be estimated at 4 billion US\$. An estimate indicated in Table 1. is based on provided data of different ferrite grade ferric oxide shipments as well as other sources [1].

Similar forecasts have been presented in the past on occasion of ICF 3 & ICF 5 conferences [2], [3]. Due to the fact, that the production of ferrites is rather labour intensive, substantial shifting of the production of ferrite cores from industrialized to developing countries has been taking place. However synthetic ferric oxides exhibiting an attractive price versus performance ratio, which are required for the production of uniform ferrite powders and cores, are to a large extent originating from integrated steel mills in industrialized countries.

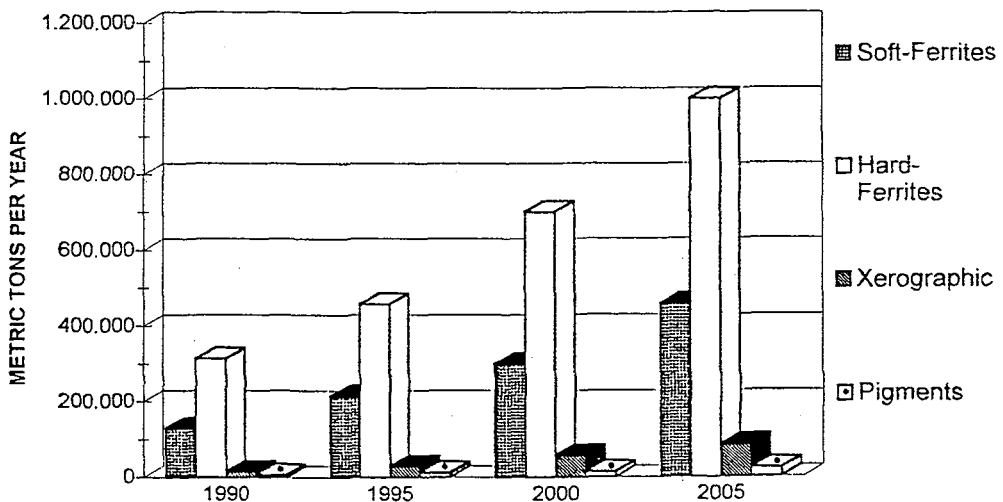


Table 1. Estimated world ferrite production in metric tons per year

2. Requirements for the Production of More Uniform Ferrite Powders by means of new Ceramic Processing Methods

For the production of uniform ferrite powders and consequently sintered ferrite cores, exhibiting uniform micro-structures within the lower micron meter range, several processes are presently being applied on an industrial scale. Over 95% of presintered ferrite powders produced make use of rotary or pendulum kilns, which employ thermal treatments up to 60 minutes, yielding undesirable partly or completely collapsed powder micro structures, which require undesirable long and expensive micronizing milling cycles, yielding consequently to a broad distribution of primary crystal sizes within presintered ferrite powder. Only extremely short presintering reactions are considered to be suitable to keep glass forming impurities uniformly distributed and yield uncollapsed ferrite powders, which are more easily to desagglomerate and yield narrow primary ferrite crystal distributions within submicron meter range.

Further developments, taking experiences with short time solid state powder reactions into consideration and avoiding thermal decomposition procedures of multicomponent metal chloride solutions, resulted in a new calcining method based on commercially available ferrite raw materials [4].

Liquid phase sintering is considered to take place in all ferrite compositions, which contain a few hundred ppm of glass forming agents or require - grain boundary engineering - with selected dopants in order to provide desired specific material properties. Working with p.a. grade or extremely high purity raw materials is for economic reasons only possible for a limited volume of ferrite products.

Large volume ferrite production is accomplished by technical purity raw materials, which are produced in huge quantities exhibiting various levels as well as fluctuations of impurities. Due considerations should be given in any publication to precisely provide data on the nature of glass forming agents down to the lower ppm range for better understanding of mechanism, which control formation of uniform micro-structures. In addition to provided analytical data all energy consuming unit processes, like thermal treatments and milling / desagglomeration procedures, should be minimized in order to provide presintered ferrite powders and finished ferrite products with more uniform distribution of impurities and primary crystal sizes.

Table 2. indicates the different crystal growth and milling / desagglomeration cycles for three ferrite powder calcining processes. The production of ferrite powders by means of rotary kiln processing is still characterized by extensive zone refining effects yielding to undesired statistical distribution of glass forming impurities, which upon subsequent milling operations may not be uniformly distributed and cause undesired duplex micro-structures upon final sintering. Ferrite powders produced by extremely short thermal treatments and subsequent milling/desagglomeration cycles, like co-roasting [5] or vertical furnace processing, yield upon precise control of processing parameters uncollapsed ferrite powders exhibiting a more uniform distribution of impurities. To be more specific, a series of process parameters, characteristic for the long processing cycle of rotary kiln calcining, have been compared with short processing cycles as provided by means of a short thermal treatment within a vertical furnace, and compiled for the production of strontium-hexaferrite powders, in Table 3. Requirements for the production of more uniform ferrite powders may be summarized as follows:

Raw Materials

- * Improve stability of ferrite raw materials impurity levels & reduce of hard agglomerates
- * Promote reduction of residual salts & increase and stabilize specific surface area data (+/- 0.25 m²/g)
- * Avoid milling equipment, which is useless to desintegrate hard agglomerates
- * Use binder systems, which burn-off free of residues & abolish inappropriate measuring technics
- * Avoid ferrite raw materials exhibiting collapsed powder microstructures

Thermal Processing

- * Use extremely short & precisely controlled presintering technics for ferrite powder production
- * Avoid production of powders exhibiting collapsed powder microstructures
- * Further reduce metal salts to less than 100 ppm
- * Consider indirect heating technics for top of the line ferrite powders
- * Control the powder cooling cycle precisely & provide rapid quality adjustment and control features

Subsequent Ferrite Powder Processing

- * Optimization of short desagglomeration cycles within the submicron meter range

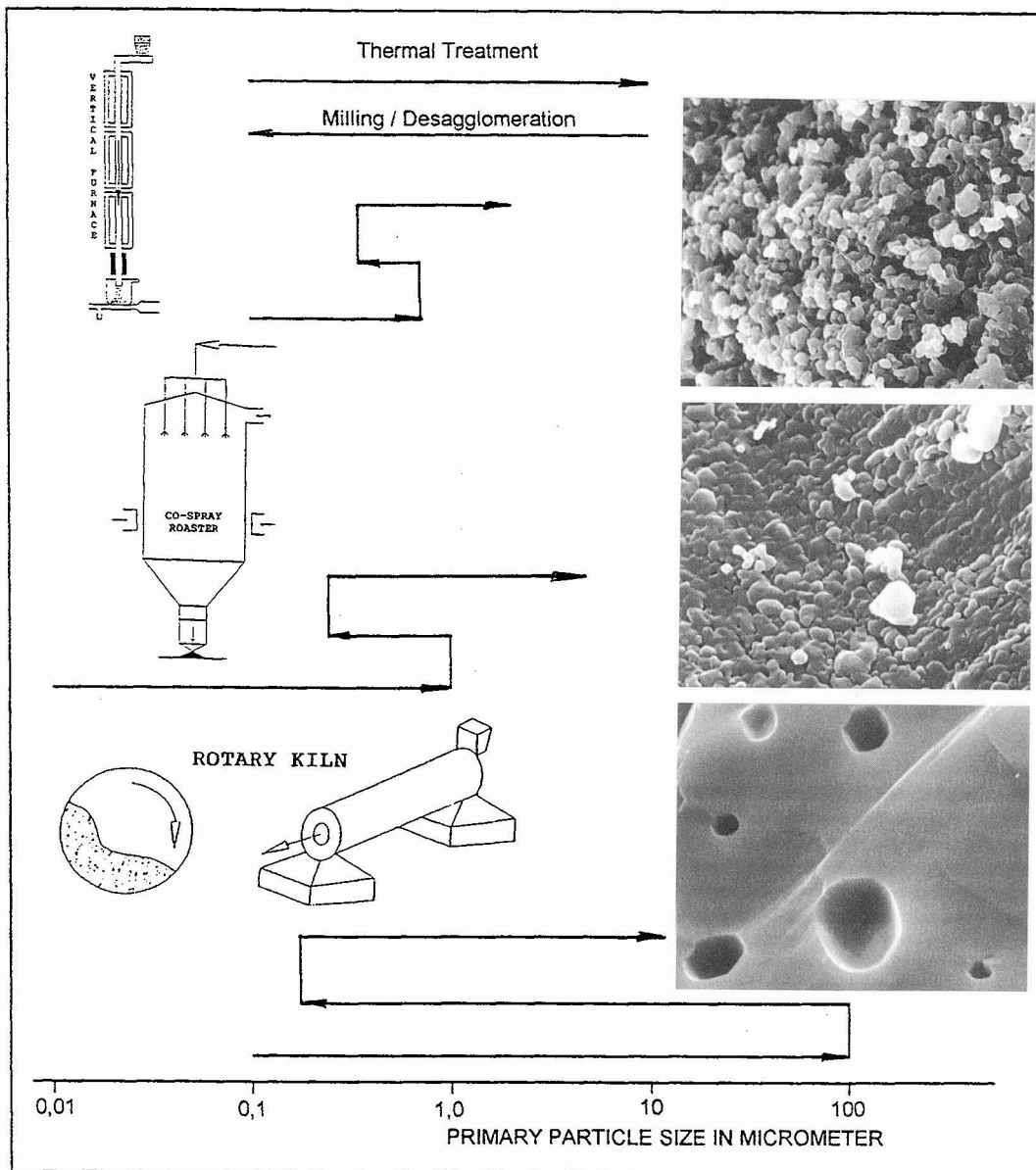


Table 3. Different primary crystal growth and milling/desagglomeration cycles characteristic for three different ferrite powder calcining processes; (1) Rotary Kiln, (2) Co-Spray Roasting and (3) Vertical Furnace
10 μ m

Conclusions

There are substantial possibilities to further improve the performance of finished ferrite products by improving a series of unit ceramic processes and operating parameters related to ferrite raw material properties, short thermal treatments, subsequent desagglomeration, granule production, or slurry processing, pressing as well as final processing steps. Substantial cost savings and quality improvements will become available as soon as more metallurgical and/or chemical industries make use of by-product ferric oxides and produce more precisely engineered presintered ferrite powders on a large scale.

FERRITE POWDER-S6F PROCESS PARAMETERS	ROTARY KILN PROCESS	VERTICAL FURNACE PROCESS
Raw Materials		
-synthetic	yes	yes
-natural	yes	no
Molar Ratio SrO.6Fe₂O₃	5.3 - 5.7	5.8 - 6.1
Raw Material Feed		
-granules, unclassified	yes	no
-granules, classified	not usual	yes
-slurry	yes	no
-granules size	3.0 - 7.0 mm	150 +/- 50 µm
Thermal Treatment		
- granule explosion	observed	no
-explosion of slurry	observed	not applicable
-dust formation	yes	no
-green granule softening	yes	no
-lixotropic effects	yes	no
-moisture content	15 - 30 wt. %	0.2 - 0.5 wt. %
-local concentration of impurities	yes	no
-general temperature range	700 - 1750°C	700 - 1800°C
-temperature range SF6	1200 - 1350°C	1050 - 1200°C
-temperature control range	+/- 20°C	+/- 2.5°C
-change of process parameters(thermal)	slow	fast
-retention time in seconds	1500 - 3000	1.5 - 3.0
-change of temperature profile	within limits	very flexible
-temperature difference at feed material upon heating	large	uniform
-crystal neck growth control	not possible	adjustable
-clinker formation	very usual	only on purpose
-specific surface control (BET)	not possible	precise
-self sintering of selected raw materials	frequently	not possible
-desired change of product	very time consuming	flexible and very fast
-precisely control of microstructure	difficult	adjustable
-primary particle size distribution	broad	narrow
-reduction of volatile matter	limited	excellent
-thermal shielding of feed material	yes	no
-material flow	rolling	quasi free falling
-control of molar composition	fair	excellent
-residual salts removal	good	excellent
-losses of metal oxides with higher vapour pressures	high	very little
-quality adjustments during production and control cycle	very time consuming	very fast
Subsequent Milling and/or Desagglomeration		
-time required	standard	very short
-fines	substantial	limited, reduced
-primary particle size distribution	broad	narrow

Table 4. Process parameters and related presintered ferrite powder properties (SrO.6Fe₂O₃) connected with long (rotary kiln process) and short (vertical furnace process) term thermal processing cycles

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