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ORIGINAL ARTICLE

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Definitive experimental evidence for *Microwave Effects*: radically new effects of separated E and H fields, such as decrystallization of oxides in seconds

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Abstract Inspite of overwhelming evidence showing that a variety of solid state reactions were radically different when performed in a multimode microwave chamber compared to conventional heating some still question whether there is direct link to the radiation field.

The present study has been conducted, for the first time ever, in a single mode TE_{103} cavity at 2.45 GHz. A large variety of materials have been reacted as ≈1 cm ϕ, 2 mm thick samples within the same cavity, at the positions of the E field and H field maxima. Enormous differences are shown to occur for certain families of common ceramic phases depending only on which of the E or H fields were used.

The most extraordinary effects and sharpest differences (noted to date) between reactions at the E and H nodes have been found in ferroic oxides. Phases such as Fe_3O_4 or binary compounds such as $BaFe_{12}O_{19}$ are rendered non-crystalline to XRD in a few seconds in the H field, although they show no bulk evidence for melting. The microstructures are unique, showing smooth glass-like regions with regular waves parallel to each other. In the E field node the identical pellet components react completely and form large euhedral crystals of a single phase. The phenomenon of de-crystallization or formation of nano-glasses was confirmed for all the 3d ferrite phases. The magnetic properties of these decrystallized ferro-magnetic phases also show remarkable changes from the original very hard, transforming to very soft, magnets.

Keywords H field · E field · $BaFe_{12}O_{19}$ · Fe_3O_4 · Ferrites · Decrystallization (“glass” formation) · Magnetic properties

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Introduction and Relation to Previous Work

The debate about the existence of microwave effects

The facts about microwave sintering of ceramics (such as Al_2O_3, spinel etc.) and semiconductors such as Si_3N_4 and SiC, have been reported in several dozens of papers. The reader is referred to reviews by Sutton [1], Clarke and Sutton [2], Roy et al [3–5] that provide a wealth of background information on the issue. As a general summary it can be stated that the kinetics of both *synthesis* reactions of more than one phase, and *sintering* reactions of single phases at the same (or even lower) temperatures are increased by 2 to 3 orders of magnitude or more by substituting microwave radiation for conventional heating.

Table 1 [6] shows the startling differences in the synthesis of $BaTiO_3$, one of the world’s most used high tech ceramic phases. A glance will show that in the conventional heating used worldwide it takes several hours to get a single phase $BaTiO_3$, and even after 10 hours only $BaTi_2O_4$ is visible to XRD. In contrast, single phase $BaTiO_3$ is completely crytallized in 5 minutes in a microwave field; and in "0” minutes at 250 °C one observes reaction products – including $BaTiO_3$, and α sequence of phases which is totally different from conventional processing.

Yet the question is still raised [3]: Are there any non-thermal causes for such effects? As though the increase in kinetics and total change in reaction paths can be explained in any other way. The real unknown, of course, is by what mechanisms the microwave energy affects the reactivity of the solid phases. One contribution to this question was provided by the work of Peelamedu et al [7, 8]. They showed that, for example, in the reaction of Y_2O_3 with Fe_3O_4 as in $BaCO_3$ and TiO_{2-x} the mechanism involved what they termed *anisothermal* reactions. That is, in a microwave field huge differences in temperatures are created, and maintained for some time, between the two reacting phases, i.e., the phase mixture is being processed (Fig. 1) under *anisothermal reaction* conditions. Such a condition is unprecedented in the 10,000 years of

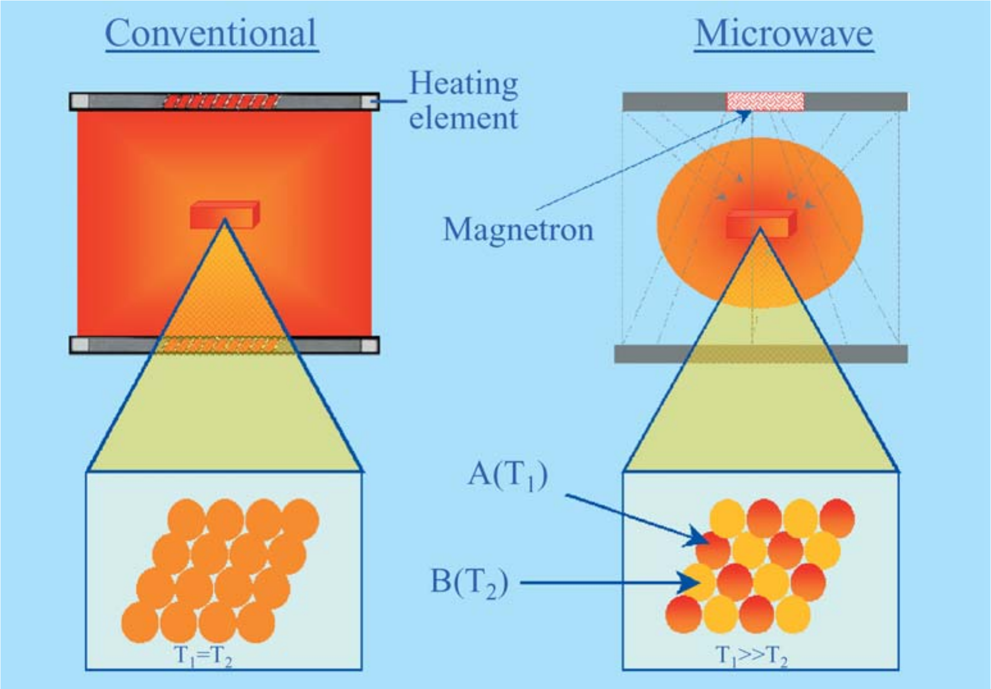

Fig. 1 Schematic diagram showing the heating behavior of a powder mixture containing both low and high absorbing powders in a microwave cavity

Table 1 XRD intensities of conventional and microwave reacted $BaCO_3$+TiO_2 powder mixture

CONVENTIONAL ($BaCO_3$+TiO_2)					MICROWAVE($BaCO_3$+TiO_{2-x})				
Temp/Soak	$BaCO_3$	TiO_2	$BaTiO_3$	Ba_2TiO_4	Temp/Soak	$BaCO_3$	TiO_2	H-$BaTiO_3$	T-$BaTiO_3$
900 °C/15 min	100	99	–	9	250 °C/0 min	87	100	7	–
900 °C/60 min	98	100	–	30	400 °C/0 min	52	45	79	12
950 °C/30 min	100	88	18	25	500 °C/1 min	22	18	100	44
950 °C/60 min	82	100	20	30	600 °C/5 min	24	28	63	100
1000 °C/30 min	67	82	45	100	700 °C/5 min	9	12	23	100
1050 °C/30 min	72	71	83	100	900 °C/5 min	–	2	–	100

the human use of thermally activated reactions to make materials. It opens up a new field of reaction mechanisms and kinetics.

This paper deals with a different and essentially new experimental approach to the problem: the use of single mode cavities to separate the electric and magnetic fields at these microwave frequencies and studying the interaction of the separate E and H fields with matter. In our first paper on these differences between E and H fields [9], we dealt only with the very different energy absorption by the representative inorganic materials in the E and H fields – metals, ceramics and semi-insulators.

Contrary to virtually the entire theoretical literature on the subject of microwave interaction with matter, which is based on the assumption that heating in a microwave furnace occurs due to losses with the electric vector alone, we found that the magnetic field was an equally or even more important part of the energy loss mechanism for many materials. In the present study, we have (studied for the first time, as far as we can determine) reacted and sintered materials in what is essentially pure E, or pure H field [10]. There is one paper [11] in the literature in which the authors showed the differences in reaction in a ceramic ferroelectric composition. They used a rod of material extending part of the distance from the electric node towards the magnetic node and showed differences in reaction along the rod. Our results obtained in the separated fields so far go beyond heating, or sintering, into the most unexpected phase changes, which we can attribute to the different reaction to E and H fields.

There is also only one relevant paper on microwave reactions with this same family of materials. In personal communications, and by exchange visits to each others' laboratories by Professor T. Endo of Tohoku University and the senior author, Professor Roy, it was found that Endo and Kimura [12] had exposed a series of pellets (≈2 cm long, ≈1 cm ϕ) of ferrites to 28 GHz mulitmode radiation at about 2–6 kw input power. The results were most unexpected. All the ferrites were rendered non-crystalline (NC≈glassy). Moreover, they reported that these noncrystalline phases were quite stable and did not crystallize for hours at ≈1000 °C but did crystalline on grinding. Hence, this family of phases – the ferruginous spinels and magneto plumbites – offered an interesting opportunity to examine the mechanism for this uniquely new route to non-crystalline NC or "glassy" phase formation.

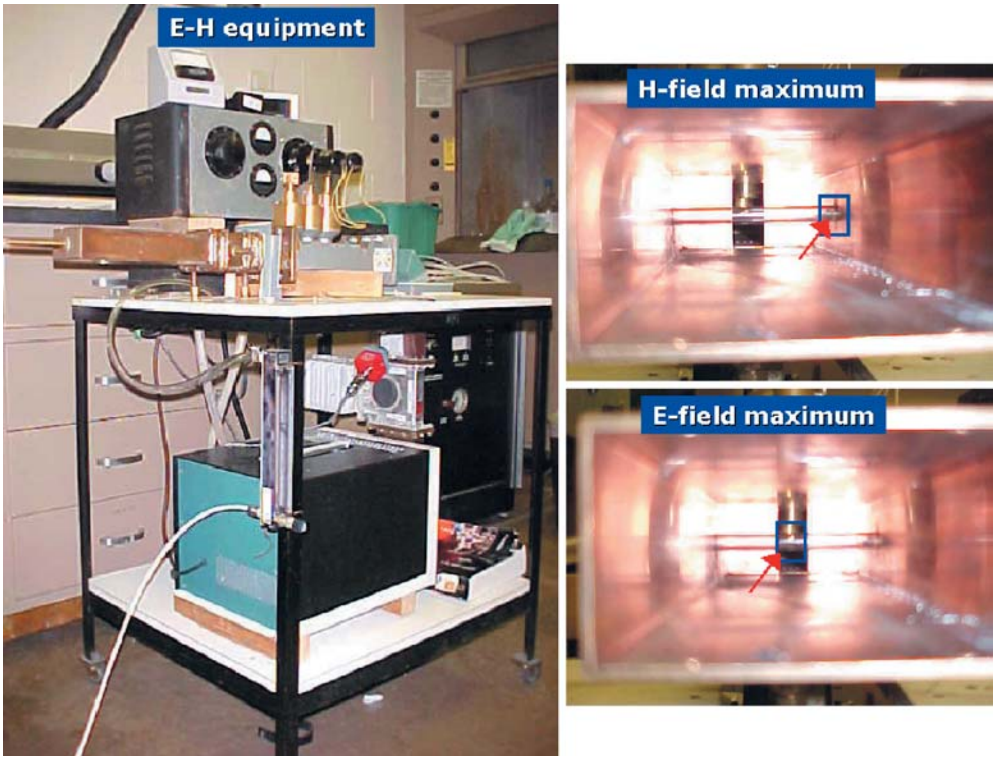

Fig. 2 *Left*: A 900 W, 2.45 GHz single mode TE_{103} microwave cavity used for E and H field heating. *Right*: The typical cavity is the rectangular copper box in the left figure. Inside it one can see the E and H field heating positions inside the silica glass tube for atmosphere control

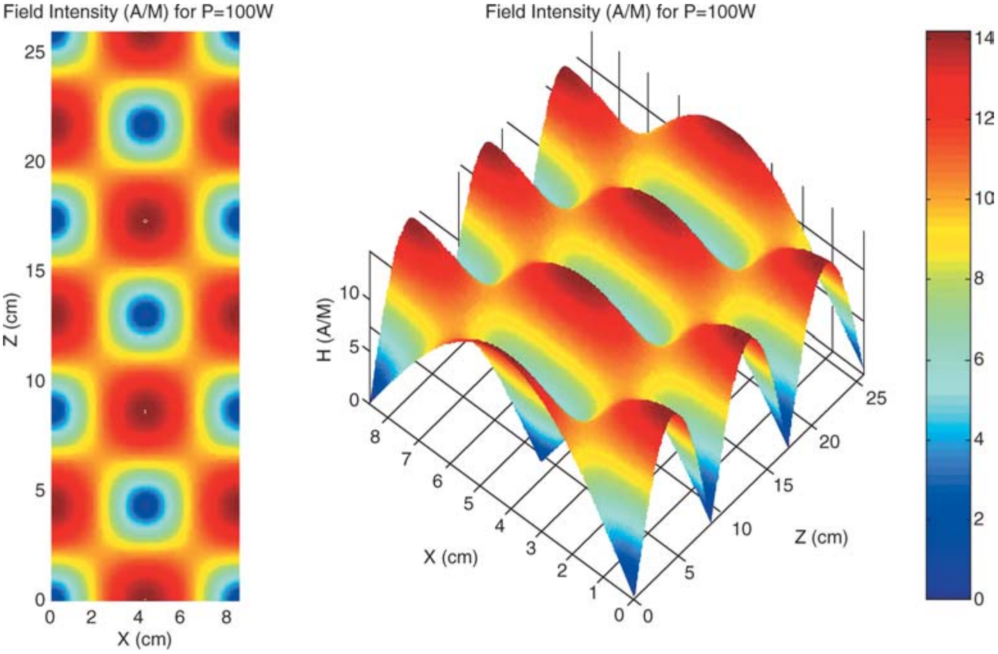

Fig. 3 2D and 3D distributions of E and H field intensity inside the microwave cavity

Experimental

Materials Studied

The materials used in this study include the principal commercial oxide ferro-magnets of industry – the ferrites, both hard and soft – and a few other oxides of 3d elements. [1]

Apparatus

Figure 2 shows the experimental set-up with a 900 W, 2.45 GHz magnetron powered generator and the locally built applicator for a single mode TE_{103} cavity. Also shown is the inside of the cavity with the location of the two pellets to be exposed to E and H fields, respectively.

Figure 3 shows the idealized distribution of the fields and the 3 dimensional distribution of the intensities of E and H fields inside the cavity. Calculations of the magnetic field intensity at the H maximum for an input power of ≈750 watts gave a value of approximately 0.5 oersteds at 2.45 GHz.

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[1] Since the sample will of course distort the field distribution, simple calculations were made using the size and mass of our samples and the size of the cavity. They show that the samples were indeed exposed to *reasonably pure* E or H fields, within several percent.

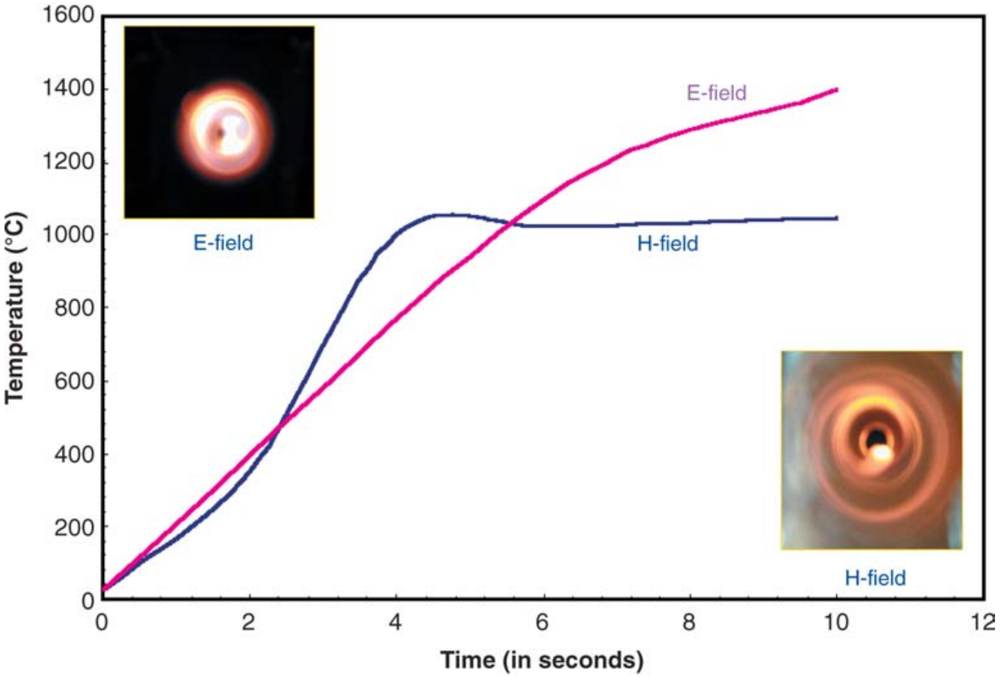


Fig. 4 Heating t-t profile of Fe_3O_4 in electric and magnetic field using a single wavelength IR pyrometer and e=0.75

Sample preparation

The powders – both two phase and single phase were not specially selected. They were made from reagent grade chemicals, ground in an agate mortar and pestle and cold isostatically pressed (CIP) at about 200 M Pa pressure. The size range of the starting powders were typically in the 0.1–2 μm range.

The pellets were set in the E and H maximum sites as desired. The power was turned on for the desired period from 5 seconds to a few minutes. Temperatures were read by a single wavelength infrared pyrometer using appropriate emissivity values chosen from the literature. For example, the time-temperature heating profile of Fe_3O_4 in electric and magnetic field using a single wavelength IR pyrometer with e = 0.75, are shown in Fig. 4.

These are the temperatures reported in the data tables, albeit we are not sure how such temperatures may correspond to say a small element say <1 mm^3 in the center of the pellets. Exposure time was counted using a stop watch.

Reaction of binary mixtures

Pellets made of $BaCO_3 + 4Fe_3O_4$ were used with Fe_3O_4 instead of Fe_2O_3 supplying the iron. This is because from previous work [7] we had established that stoichiometric Fe_2O_3 does not couple to 2.45 GHz radiation. This is an interesting rare example-among simple oxides- of the frequency dependence of the absorption of the radiation. Conversely, Kimura et al [13] found that Fe_2O_3 couples very effectively at 28 GHz while Fe_3O_4 does not.

Similar samples were made with $CoCO_3$, ZnO, NiO, etc., in place of $BaCO_3$. Single phase powder samples of other 3d element oxides such as MnO_2, CuO, etc., were also examined.

On turning on the power, with nominal input power of 300, 500, 700 watts, the temperatures of the H field pellets rises to ~1000 °C in about 10 seconds. In the case of H-field heated Fe_3O_4, the maximum temperature is ~1100 °C in 10 s whereas in the E-field site the temperatures are considerably higher, and reach 1400 °C in some cases at the highest power. Arcing is sometimes observed.

Shutting off the power causes the very modest *quenching* involved. Cooling times to below 100 °C probably are in the range of ~1–2 minutes giving a quench rate of say 10 °C/s (compared to typical splat quenching rates of 10^6 °C/s).

Results

Binary Mixtures

Figure 5 shows the scanning electron micrograph (SEM) of the top of the pellet of the $BaCO_3 + 4Fe_3O_4$ unreacted mixture. It clearly shows 0.1–2 μm size particles of $BaCO_3$ mixed with similar size particles of Fe_3O_4. The Ba and Fe maps show (in the inset) a uniform distribution of $BaCO_3$ and Fe_3O_4 particles.

After reaction the pellets occasionally crack into 2 or 3 pieces, but otherwise have not been distorted or *melted*. Very often in the H field the surfaces appear somewhat shiny. This is in radical contrast to similar materials e.g. a YBC superconductor pellet, which melts in ~3 seconds, and flows for several centimeters inside the quartz reaction tube.

Figure 6 shows the SEM of the $BaCO_3 + 4Fe_3O_4$ reacted pellet – now chemically homogenized to $BaFe_{12}O_{19}$ in the H field. Extensive EDX and X-ray mapping analyses have been carried out to check on the compositional homogeneity. The sequence from 5 seconds to 15 seconds to 90 seconds suggests an immediate reaction to the expected hexagonal magnetoplumbite morphology, which then recrystallizes into bigger units (15 seconds) and then “flows” – possibly due to lower viscosities at higher temperatures.

Figure 7 shows the SEM of an identical pellet in the same apparatus for the same time, in the E field. The differences are extraordinary; the well-crystallized euhedral morphology being formed *in seconds* is totally unheard of in ceramic synthesis.

Figure 8 shows the X-ray diffraction (XRD) pattern of $BaCO_3 + 4Fe_3O_4$ after various durations of H-field exposure. This is the product we call “decrystallized”

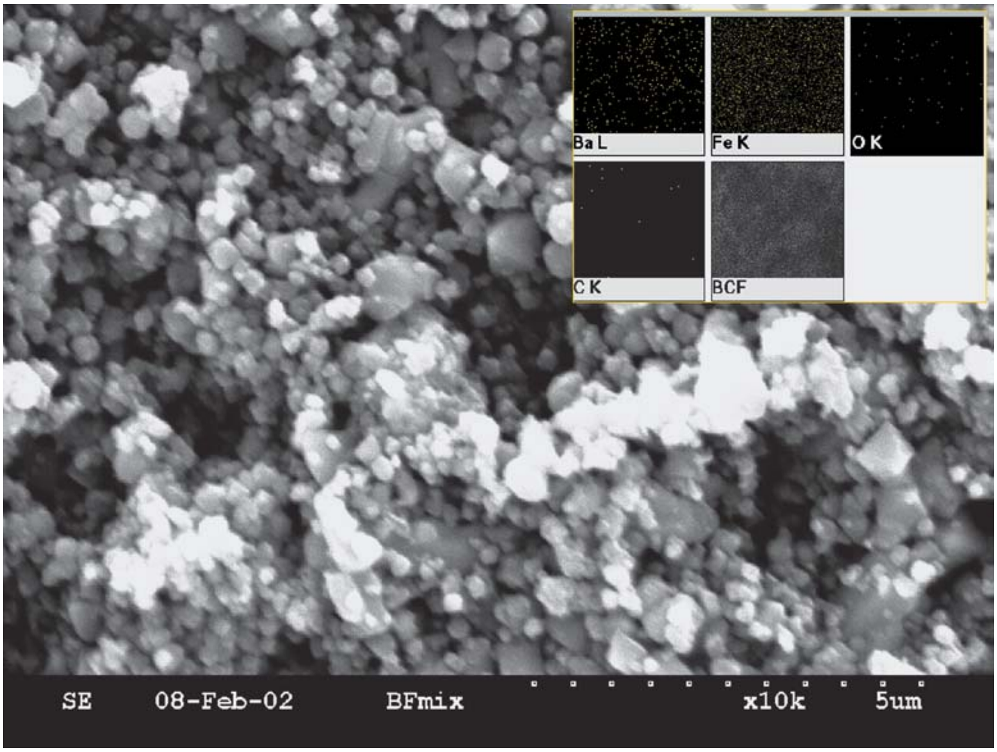


Fig. 5 Scanning electron micrograph (SEM) of $BaCO_3$+4Fe_3O_4 starting mixture showing micron and submicron sized particles. (Inset) X-ray maps of the powder mixture

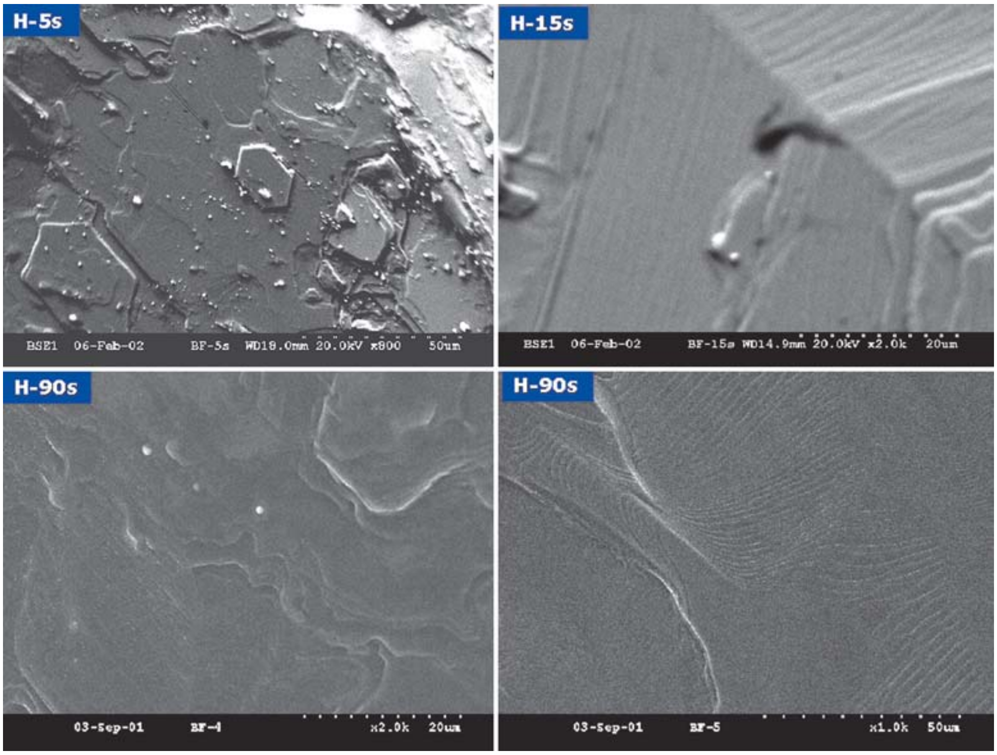


Fig. 6 Sequence of SEM microstructures of $BaFe_{12}O_{19}$ obtained after exposure of $BaCO_3$+4Fe_3O_4 mixture to the H-field

barium ferrite. It cannot be properly classified as a glass, since there is no evidence for melting (macro flowing) or even attaining temperatures near its melting point.

In the E-field the XRD pattern shows (Fig. 9) complete reaction with formation of only crystalline $BaFe_{12}O_{19}$ after 90 seconds. In both H (left) and E (right) fields, they react (with complete CO_2 loss) in a few seconds. In the case of the H field, the data show that the two crystalline phases have been decrystallized and the resulting *compound* is also decrystallized. The sample is

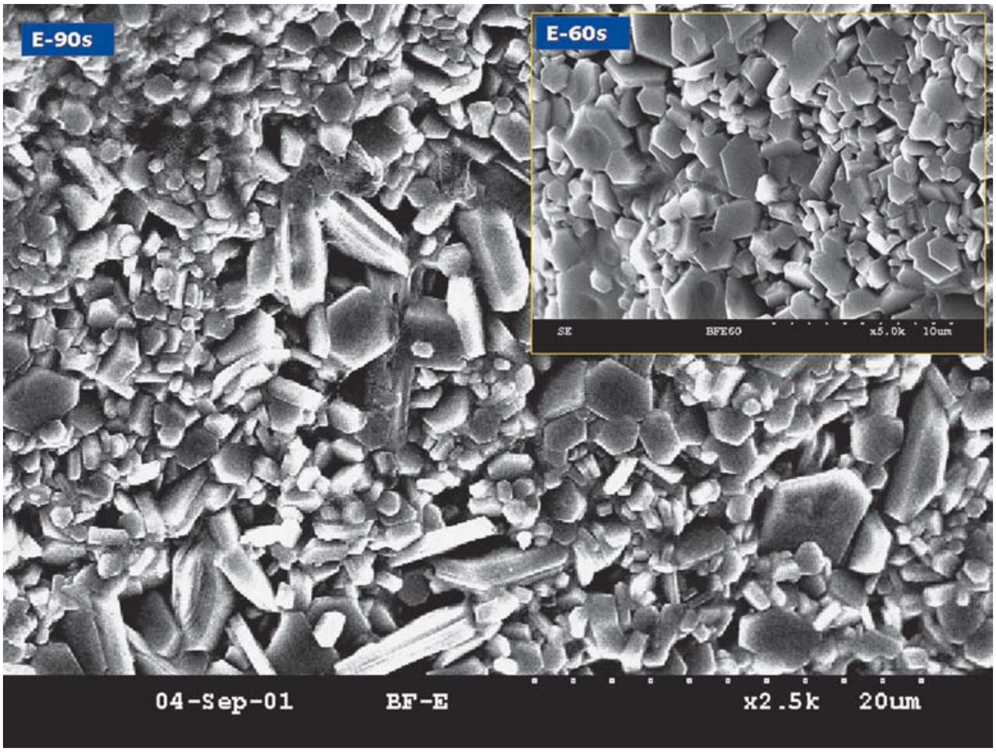


Fig. 7 Sequence of microstructures of $BaFe_{12}O_{19}$ obtained after exposure of $BaCO_3$+$4Fe_3O_4$ mixture to the E-field

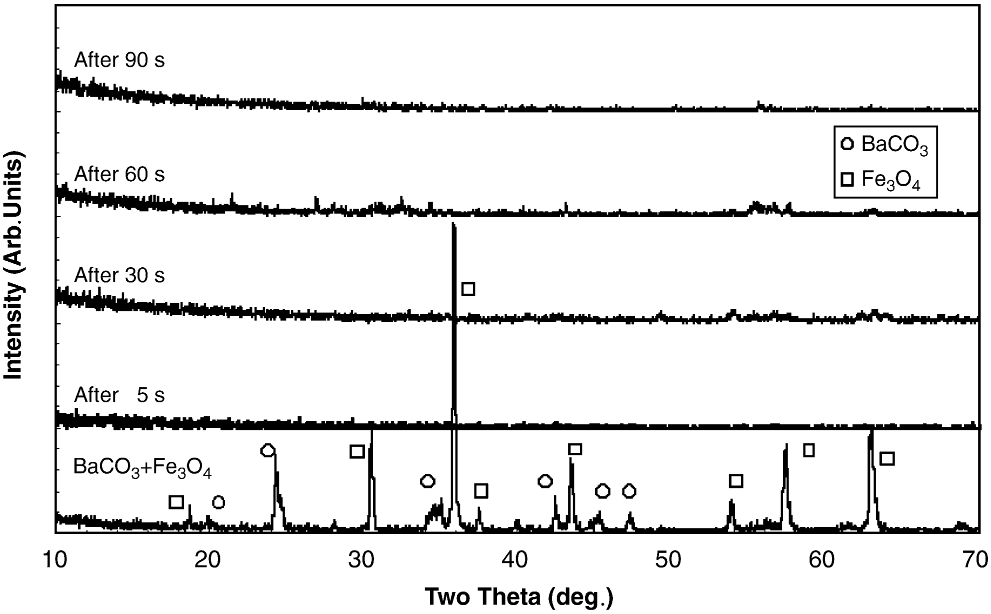


Fig. 8 Decrystallization of ferrites (such as $FeFe_2O_4$, $NiFe_2O_4$, $CoFe_2O_4$, etc.) proven conclusively from x-ray data shown for $BaFe_{12}O_{19}$, in a 2.45 GHz magnetic field. The XRD patterns of normal crystalline phases are converted to the typical amorphous pattern as shown, in 5–90 seconds

fully reacted and has a glassy liquid appearance. There are no Bragg reflections at all. Moreover this is not merely at the surface, but throughout the pellet as sections perpendicular to the surface show the same XRD and SEM results. The morphology is clearly that of a liquid, but its macroscopic lack of flow behavior would suggest a viscosity greatly in excess of 10^{14}. The decrystallization obviously involved very small volumes perhaps we have “glasses” on a nanocomposite scale. But the most extraordinary data appear in the SEM of the H field samples. We note again that no external bulk evidence of melting is present. (These liquids are very fluid at their melting points).

But Fig. 10 shows the smooth glassy appearance. The unique and always present feature are the periodic soliton-like waves throughout the structure. After 5 seconds (see inset), there is more structure with sharply spaced wavelets. After 90 seconds, the wave heights seem to be smoothed out – perhaps due to lower viscosity at higher temperatures. The periodicity is in the 1–5 μm range.

The arrow on Fig. 11 shows a kind of pile up of waves. These wave packets suggest the formation of a

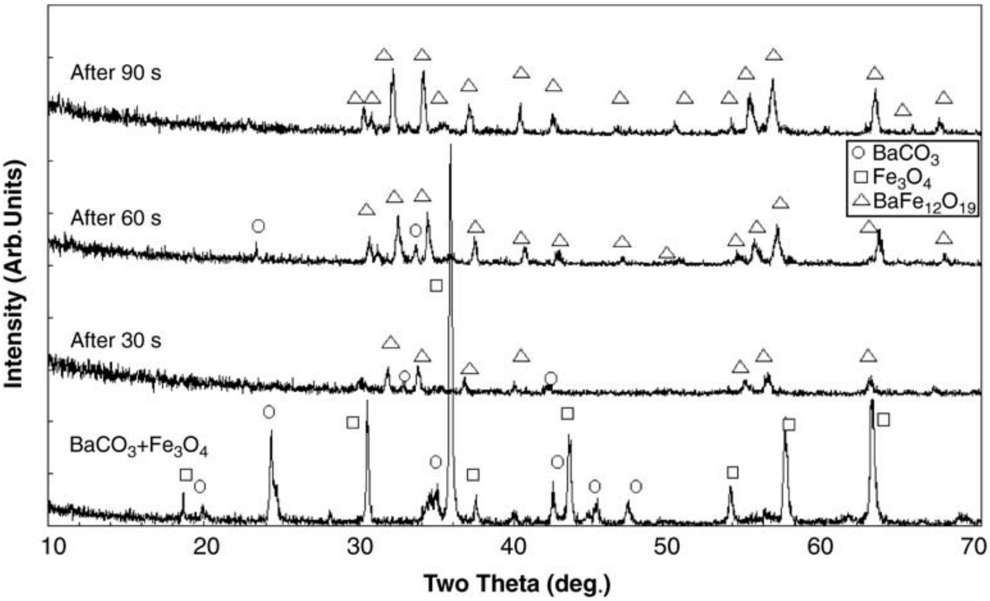


Fig. 9 XRD patterns of E-field heated $BaCO_3+4Fe_3O_4$ mixtures showing crystallization of $BaFe_{12}O_{19}$ in 30 seconds

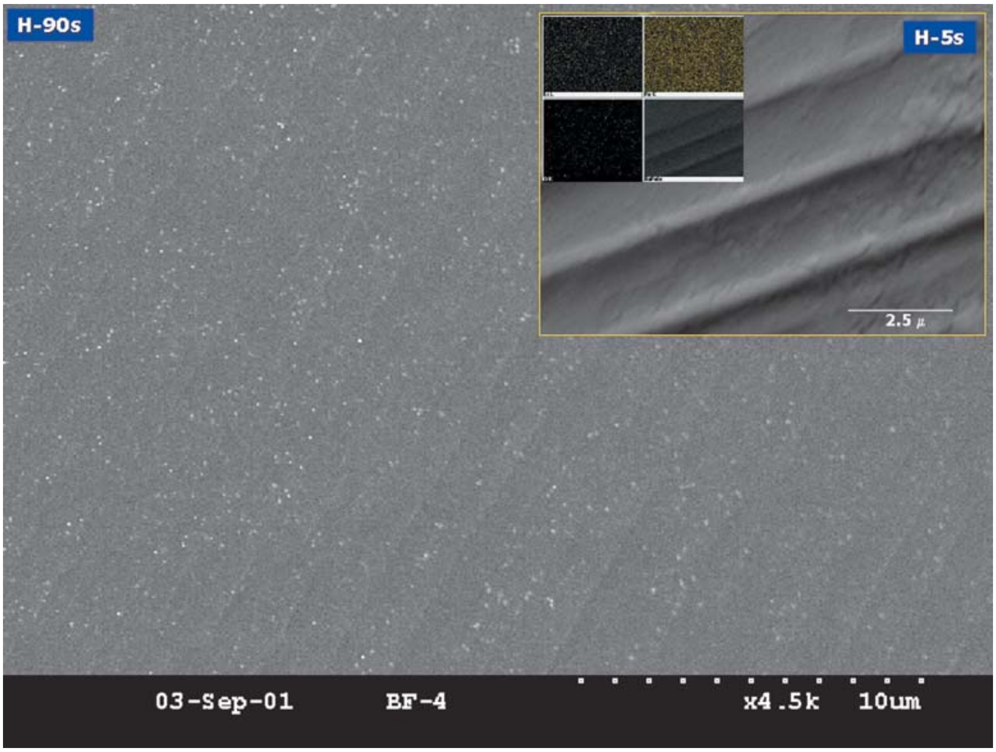


Fig. 10 SEM microstructure of $BaFe_{12}O_{19}$ obtained after 90 seconds of exposure of $BaCO_3+4Fe_3O_4$ mixture to the H-field, showing the typical glassy pattern. The large inset shows the decrystallized structure and the smaller inset shows that the chemistry has been homogenized

fluid of some kind. Yet the 'ghosts' of hexagonal units remain.

Figure 12 shows XRD pattern of polycrystalline Fe_3O_4 powder reacted in a H-field. Just after 60 seconds of H-field exposure, Fe_3O_4 has been decrystallized completely.

The SEM of the decrystallized sample is given in Fig. 13, which appears very much like decrystallized $BaFe_{12}O_{19}$. The 60s, H-field decrystallized Fe_3O_4 sample was randomly fractured and SEM examination was carried out on the fractured surfaces.

Cross-sectional microstructure (Fig. 14) failed to reveal any visible grains, grain boundary morphology, thus confirming that 'decrystallization' occurs in bulk rather than just on the surface. The pellet reacted in the E field could not be more different.

The XRD data show the oxidation to a well-crystallized Fe_2O_3 phase in just 30 seconds (Fig. 15).

The SEM of the same sample is shown in Fig. 16. SEM of E-field reacted $BaFe_{12}O_{19}$ (Fig. 7) shows well formed euhedral hexagonal plates. The typical coercivi-

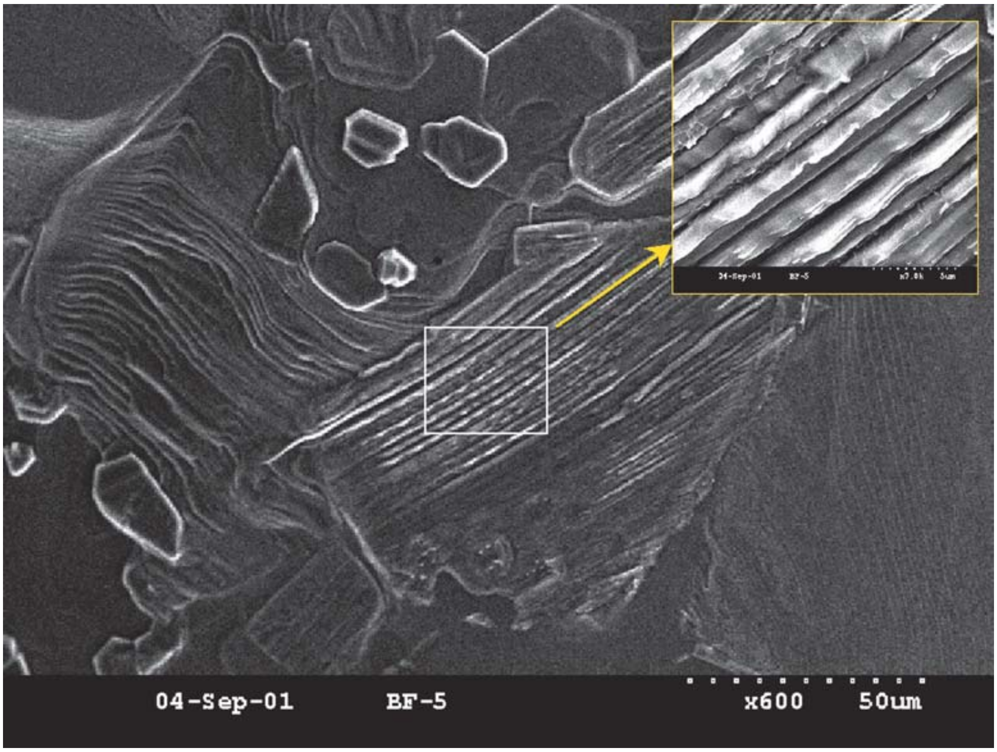


Fig. 11 Microstructure of $BaFe_{12}O_{19}$ obtained after 90 seconds in H-field showing unusual striations across the sample

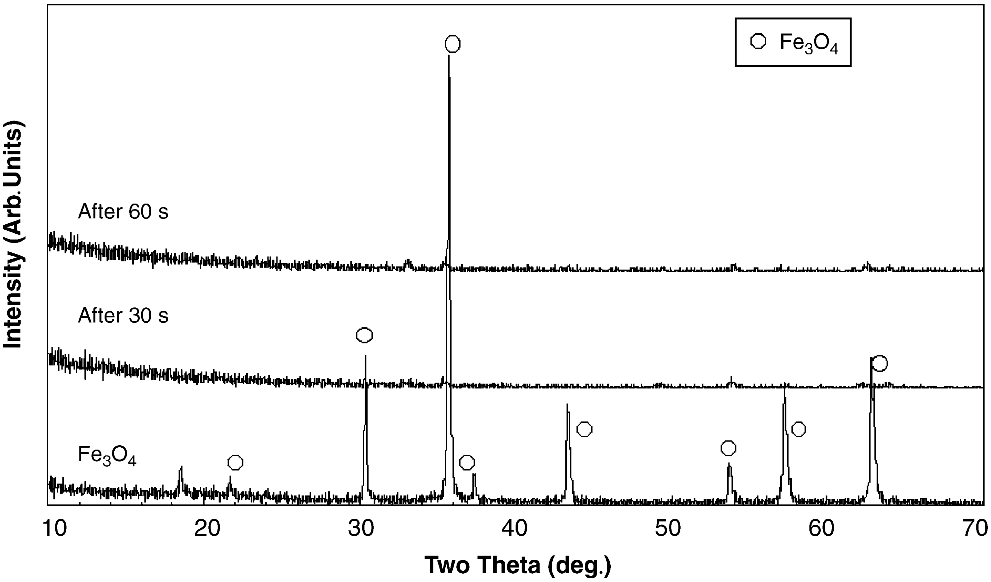


Fig. 12 XRD pattern of Fe_3O_4 powder exhibiting the decrystallization phenomenon in a microwave H-field

ties for these materials – the main stay of the ceramic magnet industry – are in the 70–80 oersteds range.

The contrast between processing such materials in the H field and E field could not be sharper – the former yielding a noncrystalline smooth solid and the latter an assemblage of well formed crystals of the expected phase – both at rates at least 10^2–10^3 times faster than in usual ceramic reaction times. It is also essential to note that in all cases *the temperatures at the E-field node are much higher than at the H-field node, although it is the latter which only shows decrystallization.*

Other ferrites: Co and Zn

Endo et al. [12] had reported that all the ferrites could be decrytallized at various power levels at 28 GHz which were an inverse function of their magnetostriction coefficients. We show in Fig. 17 the x-ray diffraction data on $CoFe_2O_4$, which shows its decrystallization, and its recrystallization upon grinding in mortar and pestle, confirming what Endo and Kimura showed for $NiFe_2O_4$. Our data at 2.45 GHz obviously drastically differ by being conducted in a single mode cavity, but are not complete

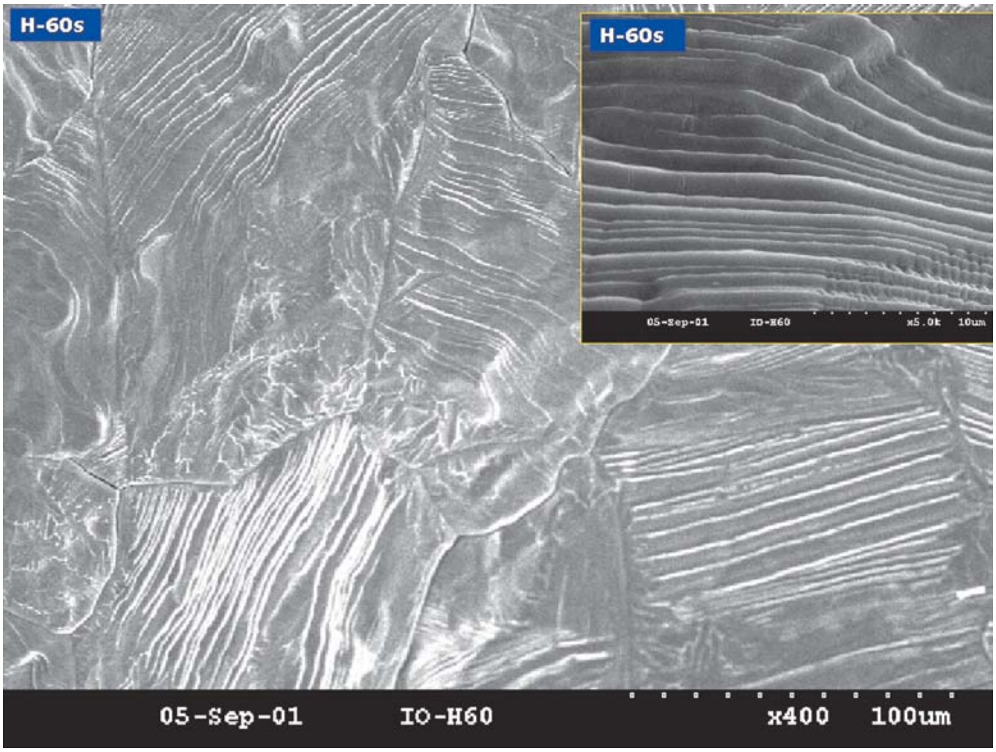


Fig. 13 SEM microstructure of Fe_3O_4 in a H-field, showing the same unusual striations as observed in $BaFe_{12}O_{19}$

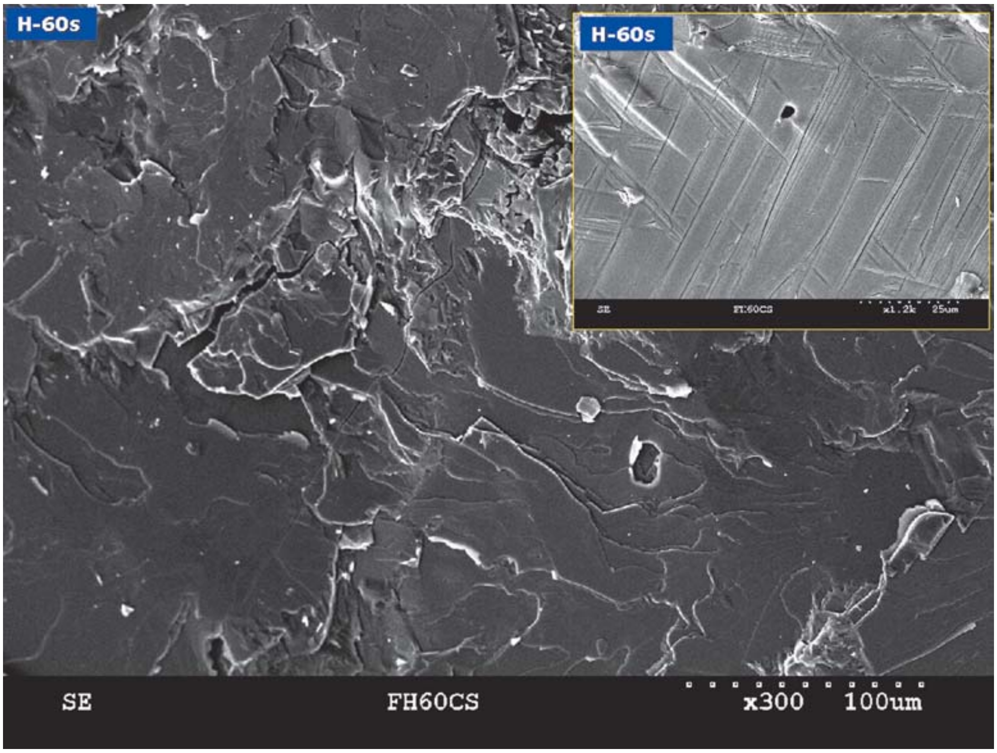


Fig. 14 Microstructure of the surface of a fractured sample of decrystallized Fe_3O_4 perpendicular to the original surface. The glassy morphology is the same, thus confirming that this is a bulk effect rather than just surfacial

enough on all phases to confirm their magnetostriction correlation. In the case of $ZnFe_2O_4$ there was a sharp break in the power – crystallinity loss at about 2 kw.

This decrystallization of $ZnFe_2O_4$, which is anti-ferromagnetic, as a function of power applied in the H field alone at 2.45 GHz is shown in Fig. 18.

The SEM of the decrystallized $ZnFe_2O_4$ (Fig. 19) show the usual periodic striations that are similar to the ones observed in Fe_3O_4 and $BaFe_{12}O_{19}$ samples.

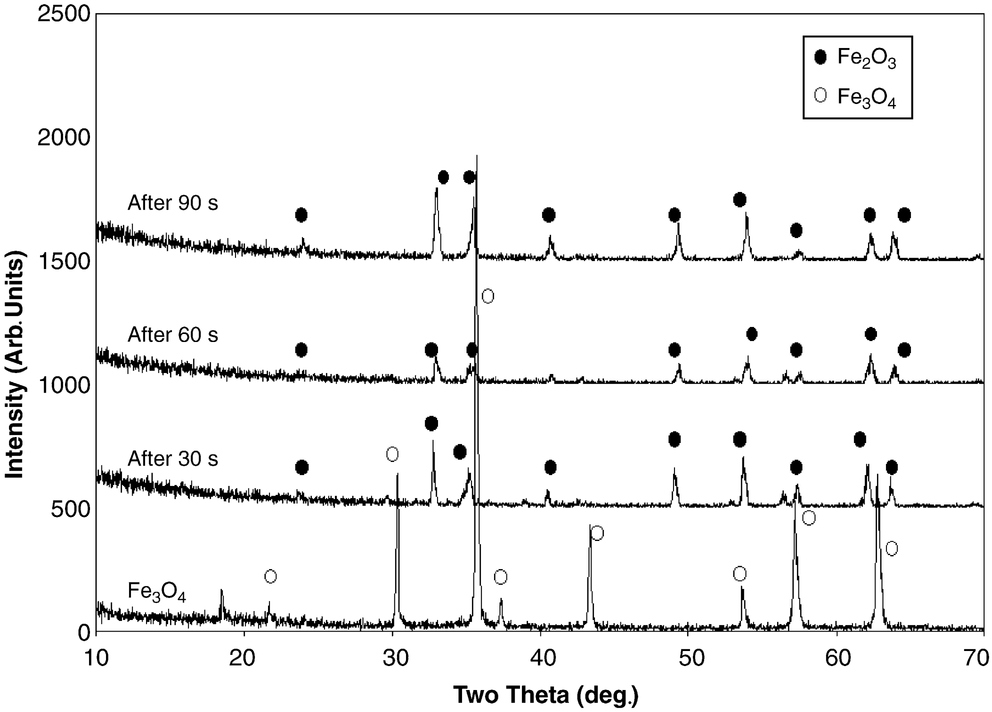


Fig. 15 XRD patterns of Fe_3O_4 powder exposed for increasing times to a microwave E-field. Notice the rapid oxidation to Fe_2O_3

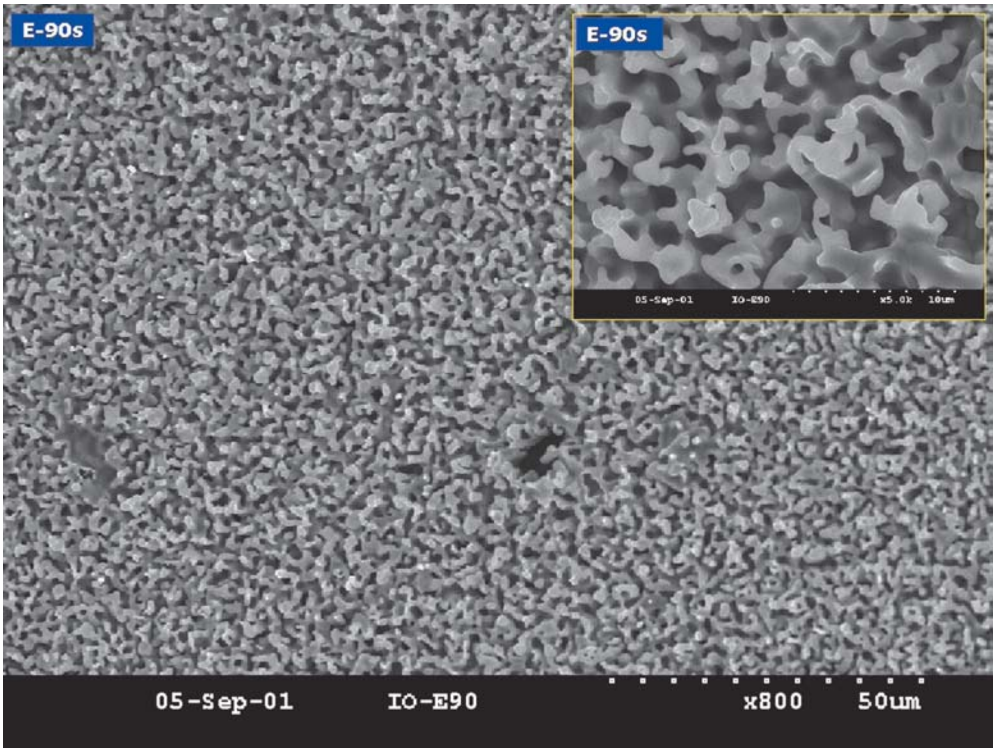


Fig. 16 Microstructure of Fe_3O_4 in an E-field, showing possible melting and oxidation but remaining fully crystalline

Magnetic Properties

The data on the magnetic properties of these materials are reported elsewhere [14] and show that while the ferrites made in the multimode chambers and E-field (in single mode) have the usual wide hysteresis loops, all the materials treated in the H-field immediately collapse to become soft-magnets. $ZnFe_2O_4$ transforms from antiferromagnetic to ferromagnetic.

Fig. 17 *Bottom*: $CoFe_2O_4$ reacted in H-field shows the usual "glass-like" pattern. *Top*: On grinding this material recrystallized to a good spinel

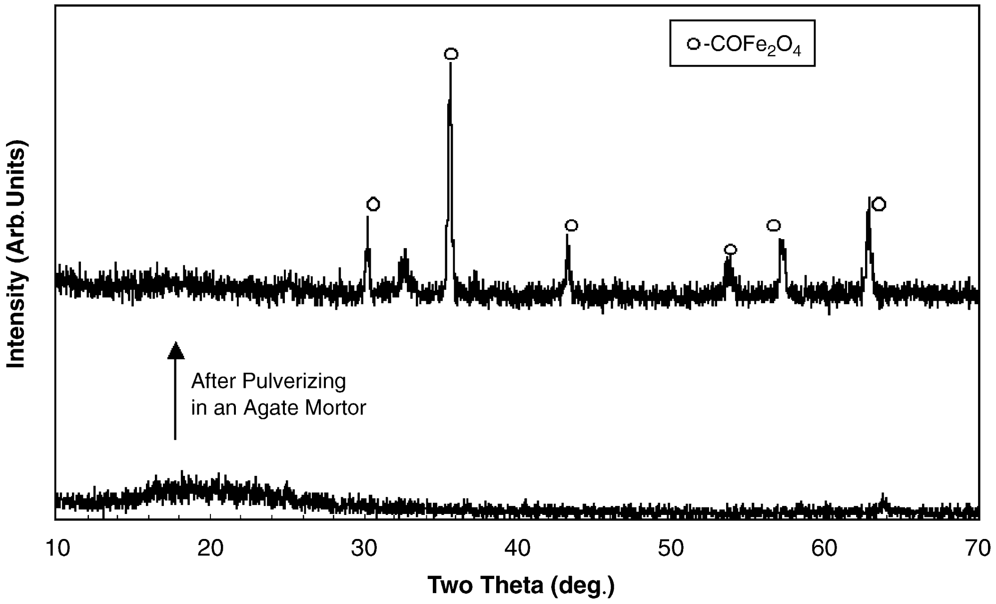
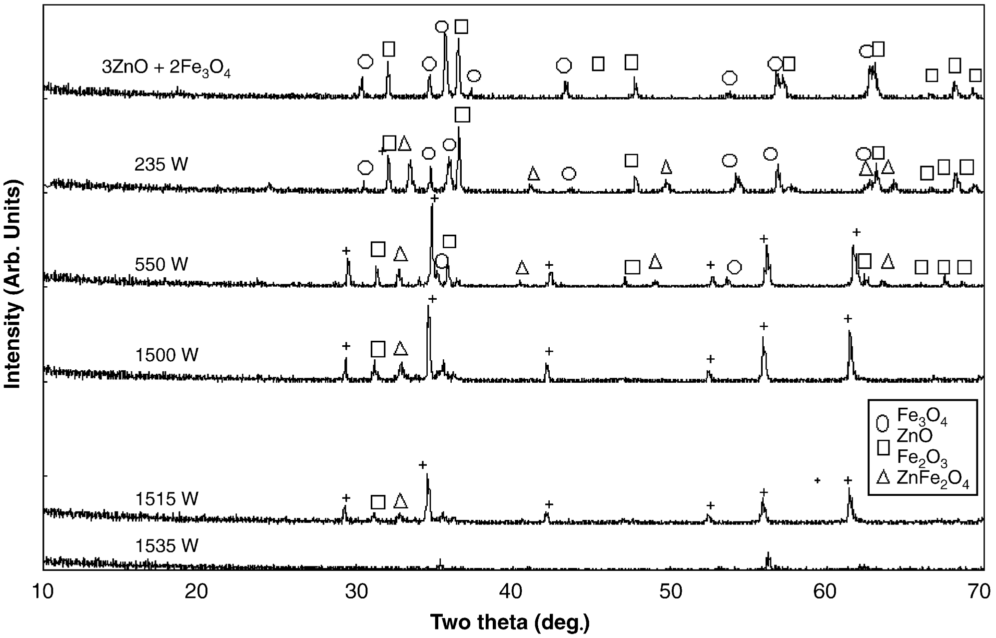


Fig. 18 XRD data showing how the decrystallization phenomenon depends on total power observed in the case of $3ZnO + 2Fe_3O_4$ mixture



Decrystallization of other non-ferrite, non-ferromagnetic oxides: CuO, MnO_2

The questions which suggest themselves include: Is the phenomenon unique to ferromagnets? Does it always require Fe atoms? These questions have been conclusively answered in the negative.

We present [15] the detailed data on the observation of decrystallization of other 3d oxides which are not ferromagnetic elsewhere [15]. As examples we show here that both CuO and MnO_2 exhibit the same phenomena. This shows that the de-crystallization phenomenon is not associated only with ferromagnetism. The SEM of the decrystallized CuO and MnO_2 is shown in Fig. 20. The most peculiar CuO microstructures have not been analyzed and repeated several times as yet but the unusual periodic stripes are seen again in the MnO_2 microstructure.

Discussion

The major findings of this work are very unexpected. So far no explanation or theory to explain the facts described, is worth much emphasis, since many unusual facts are still being accumulated. These *salient facts* are:

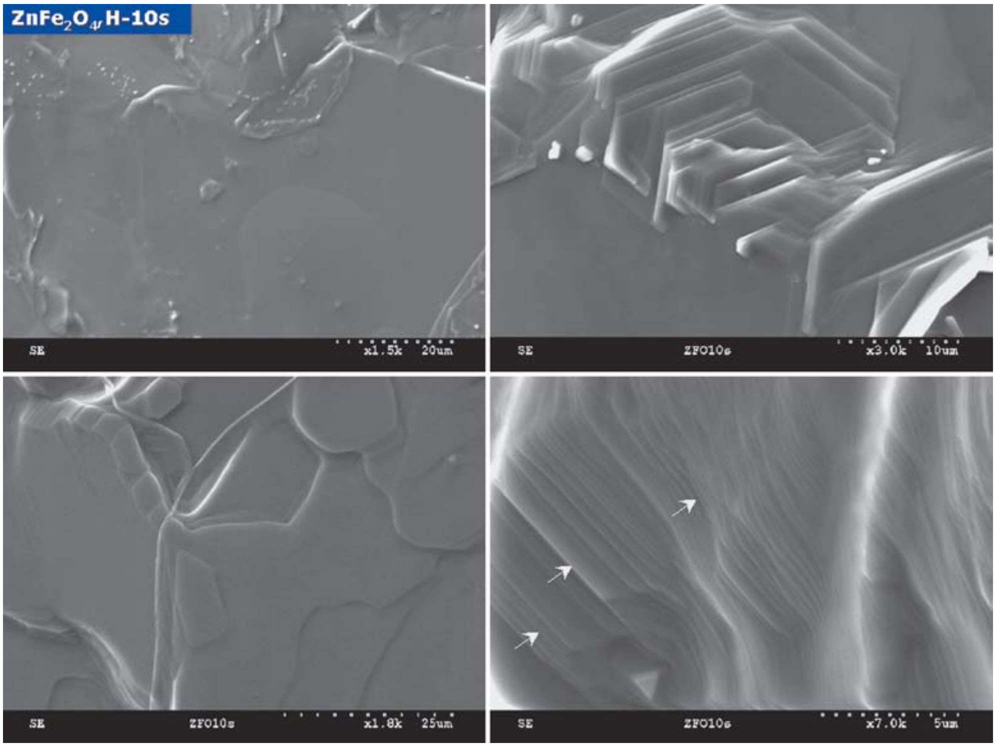


Fig. 19 SEM of decrystallized $ZnFe_2O_4$, showing step-like striations as observed in Fe_3O_4 and $BeFe_{12}O_{19}$

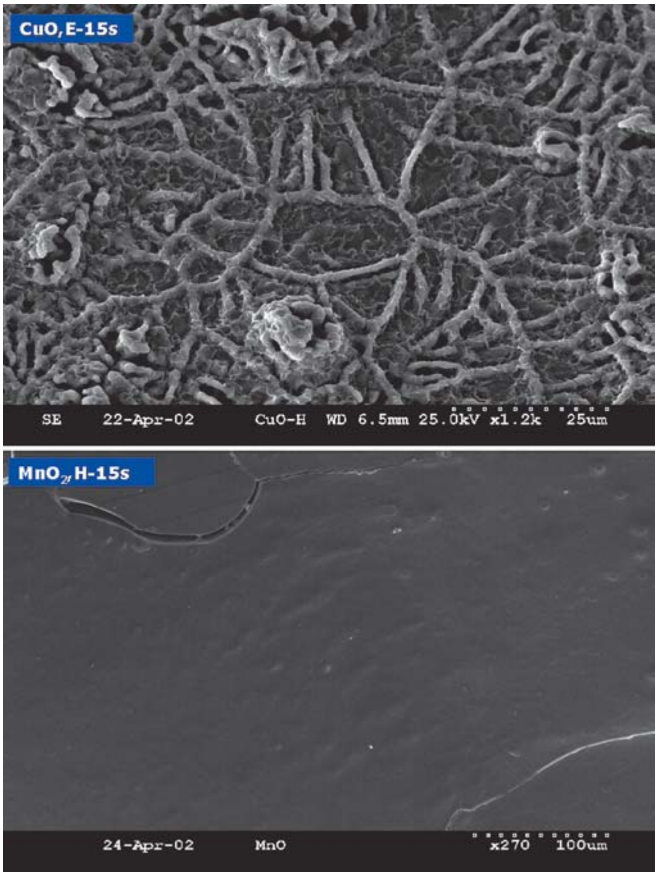


Fig. 20 Microstructure of decrystallized CuO and MnO_2. The MnO_2 exhibits the striations (faint) like many other decrystallized ferrites

1. A large number of oxides containing 3d ions, most of them ferromagnetic, undergo a solid-state phase transformation from a crystalline phase to a non-crystalline phase in a modest (≈0.5 oersted) magnetic field at 2.45 GHz in a few seconds at temperatures far below the melting point. Ferromagnetism has been ruled out as a requirement for decrystallization.
2. These decrystallized phases have no parallel in past work. They are *not* true glasses, since they have not been through the normal (*bulk*) liquid state required for being classified as glasses. The nearest analogue may be solid state decrystallized phases made by bombardment with high energy neutrons or α particles. What is so striking is the difference in energies in the radiation causing the change.
3. The presence of the remarkable wave-like patterns in the SEMs certainly indicate a liquid-like behavior on a very small scale, suggesting the action of a shock wave of some kind. Perhaps the term **nanoglasses** is not inaccurate.
4. Radical differences in interaction of the 2.45 GHz E field and the H field with ferroic oxide solids have been observed as gross differences in the phases produced, as proved by XRD and SEM of the respective products.
5. It is not possible to ignore the E and H fields as intensive variables in the thermodynamics of solids as materials scientists choose to do in analyzing phase relations in condensed matter. In virtually all such treatments one considers only pressure and temperature as affecting the free energy function, G. Thus, the standard formulations:

$$G = H - TS = U + \underline{PV} - \underline{TS} + 1/2h\nu$$
and
$$\frac{\delta G}{\delta T} = -S \quad \frac{\delta G}{\delta P} = V \tag{1}$$

The formalism for the influence of E and H on free energy has been studied by some specialists. It has been developed especially by workers interested in ferroic materials (ferroelectric and ferromagnetic) where the effects of E and H on G may be expected to be maximum, because of collective orientation effects, Newnham [16] shows one formulation as:

$$\begin{aligned}\Delta G = {} & \Delta\varepsilon_{(s)ij} + \Delta P_{(s)i}E_i + \Delta M_{(s)i}H_i + \frac{1}{2}\Delta S_{ijkl}\sigma_{ij}\sigma_{kl} \\ & + \frac{1}{2}\Delta\kappa_{ij}E_iE_j + \frac{1}{2}\Delta\chi_{ij}H_iH_j + 2\Delta d_{ijk}E_i\sigma_{jk} \\ & + 2\Delta Q_{ijk}H_i\sigma_{jk} + 2\Delta\alpha_{ij}H_iE_j\end{aligned}$$

This merely illustrates the complexity of the theoretical task ahead. This will be especially true where no special orientation states (i.e. ferroic materials) are involved.

Speculations

Endo et al. [12] offered the idea that the fact of the decrystallization was in some way related to the magnetostriction coefficient of the material, the higher the latter, the less the power needed to achieve that end.

The involvement of magnetostriction at 10^9 cycles/second as a means of destroying the positional order in the unit cell resulting from the displacement of the atoms asymmetrically in the structures, followed by a "quenching" effect of turning off the field, seems eminently reasonable, especially in the light of our key finding that it is only the magnetic field that is active in creating these new "nano-glasses". Their work was all done in a multimode field at 28 GHz and typically at considerably higher powers. However the enormously enhanced kinetics of reaction of the two phases, the speed of homogenization and the unique 'soliton'-like resulting glassy microstructure remains to be explained. A kind of local melting only on a nanoscale may be involved, since no macroflow is observed by Endo et al, or us. Yet the flow to give a smooth glass at least on a ten micron scale and most specifically the parallel wave microstructure call out for explanations.

Conclusions and Future Work

This laboratory is continuing to map out the compositional boundaries where such phase change phenomena caused by separate H and E fields can be observed. The first of special ferromagnetic *metallic* compositions show very little, but *some* change. Highly magnetostrictive metals such as Terfenol D are among the latter. The influence of frequency and power, and sample orientation towards the field etc. offer a very large playing field in studying what we now term *electromagnetic processing of materials*.

While ferromagnetism has been ruled out as an *essential* necessity for decrystallization, the required presence of d and f electrons and possibly two valence states giving rise to charge transfer or narrow d-bands may be proved by newly accumulating data.

It would appear that these extensive data establish that electromagnetic fields can make substantial contributions to the phase stability of certain groups of common solids.

It will be interesting to observe how fast and widely such a far reaching possibility – that is, the utilization of electromagnetic radiation for processing will be studied by other investigators. The experience of the senior author's introduction of sol-gel processing in 1948–56 shows that it took some 25 years to be widely studied used because most materials scientists were unfamiliar with solution chemistry. The use of magnetic and electric field processing is similarly foreign to the *experience* of a very high percentage of materials scientists. It is, for that reason also unlikely to be understood or appreciated or utilized very soon by a large number of investigators.

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