The study of correlation between microstructure of ferrites and their complex permeability spectra

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\textbf{Abstract.} Theoretical model for complex permeability of polycrystalline ferrite (based on effects arising from realities of typical microstructure of sample) is correlated with experimental data. In the case of NiZn-ferrites there is observed a close agreement with the model and the data; for samples of MnZn-ferrites this agreement is only for small-sized cores; for bigger ones the dimensional effects (dimensional resonance) appear and the model cease to be valid.

\section{1. Introduction}

One of the leader of nowadays technologies - electronics are developing very fast (according to Moore's Law) largely because of app materials and their technologies. Nevertheless there are branches, especially magnetics-based, that are lagging behind because of insufficient progress. As such, in fact, are polycrystalline ferrites (PF) that may be thought of as main high frequency magnetic materials.

One of main characteristic of ferrites at high frequencies is their complex magnetic permeability (CMP) \( \mu(f) = \mu'(f) - j\mu''(f) \), specified by its real \( \mu'(f) \) (dispersion, DCp) and imaginary \( \mu''(f) \) (absorption, ACp) components. Obviously CMP is housing large amount of information on intrinsic processes of PF. The interpretation of the data coming from these spectra has been in progress for several decades but as a rule they are only qualitative (except one specific point of the spectrum – the static permeability \( \mu(0) \)). Specifically, these interpretations as well as direct experiments show on close correlation between the features of spectrum and the microstructure (MS) of sample (within frequencies up to the ending of the most dominant range of dispersion – the large-amplitude broadband one at radiofrequencies [1]). But closer examination of this correlation is difficult principally since PF as materials are not well defined systems because of great variety of influencing factors [2].

The only known quantitative theoretical analysis of this correlation is the modelling [3] based on the realities of MS: account of effects of polycrystal grain sizes and their distribution that, after all, brings to closed-form relation for \( \mu''(f) \) which in its turn allows for \( \mu'(f) \) through the use of numerical integration of Kramers-Kronig relations (KKR) [4]. By this way gained theoretical curves for \( \mu'(f) \) and \( \mu''(f) \) provides the means of presentation of both relaxation and resonance types of MS thus indicating on
universality, at least in principle. But problems related with the details of presentation of CMP (true values of parameters, their actual correlation with MS) still remain. Answers are a matter both of experimental verification and theoretical analysis, which in this investigation is performed for samples from both of two main groups of ferrites, i.e. NiZn and MnZn ones. Within this correlation process the correctness of every used experimental $\mu(f)$ additionally is verified by KKR.

2. The essence of modelling.

As it was mentioned before the modelling of $\mu(f)$ is based on realities of MS of PF (i.e., sample is viewed as aggregate of polycrystal grains of the current size $D$, distributed by log-normal probability density function having the median $D_{med}$ and the standard deviation $\sigma_D$) as well as on assumption that these grains are forming magnetically independent elementary volumes (as evidenced, likely, by linear dependence of $\mu(0)$ on average grain size $D_a$ both by experiments [2] and theory [3]) and on hypothesis that characteristics of magnetization process within current grain $D$ resembles the ones observed in variety of real PF samples in relation to their $D_a$. Then the domain wall (DW) (or equivalent DW) in grain $D$ (without intragrain defects) acts as independent oscillator having the resonance frequency $f_0(D)$; averaging of the oscillators over the sample gives [3]:

$$
\mu''(f) = \mu''_{max} \exp \left[ - \frac{(\log f - \log f_u)^2}{2\sigma^2} \right] \tag{1}
$$

where $f_u$ and $\mu''_{max}$ are characteristics of extremum of absorption, i.e. $\mu''_{max}=\mu''(f_u)$ and $\sigma = 2\sigma_D$. This three parameters ($f_u$, $\mu''_{max}$ and $\sigma$) Eq.1 gives rise to so called symmetrical ACp (plotted along typical logf axis) which would be realizing in the case of ideal MS (when there are no intragrain defects) and giving $\sigma \approx 0.4$ [8]. However, most of the experimental ACp are considerably asymmetric. It was showed in [5] that in this case (because of action of intragrain defects) there is the need in Eq.1 to operate with two values of $\sigma$: the one $\sigma_a$ for $f \leq f_u$ and the other, $\sigma_b$ for $f \geq f_u$. The values can be obtained from [3]:

$$
\sigma_a = \sqrt{2\ln[\mu''_{max}/\mu''(f_u)]}
$$

$$
\sigma_b = \sqrt{2\ln[\mu''_{max}/\mu''(f_u)]}
$$

Fig.1. CMP of ferrite with unhomogenous MS

Fig.2. Raman spectra of MnZn and NiZn PF

Fig.3. CMP of NiZn PF with homogenous MS
in which the data from experimental ACp are inserted: \( f_a \leq f_u \) (often such as \( \mu''(f_a) \approx 0.5\mu''_{\text{max}} \)); for \( \sigma_b \), \( f_u \) is simply replaced by \( f_b > f_u \) (if for estimation of \( \sigma_a \) and \( \sigma_b \) exact half-level absorption is used, then Eq.2 simplifies to: \( \sigma_a = 0.85[\log(f_u/f_{0.5})] \), where \( f_a = f_{0.5} < f_u \) and \( f_b = f_{0.5} > f_u \). This line of the reasoning in derivation of Eq.1 shows on its claim to present \( \mu(f) \) as the intrinsic CMP of samples; nevertheless in the case of MnZn ferrites this can be obscured by dimensional resonance [6].

3. The samples and results
This research (finally aiming to either validate or disprove the proposed model) is based on experimental data from ring samples of different sizes (Table 1) and known MS (which were obtained from micrographs by the method of Saltikov [7]). Within the model there is assumed that MS is homogeneous (i.e. have the log-normal distribution) that was confirmed by all there used samples (having, Table 1, \( \sigma_{nD} \approx 0.5 \), that is the criteria for normal, homogenous MS [9]). In addition the micrographs of the ferrites (Fig.3…5) show that there are numerous defects within grains, which leads to asymmetrical CMP spectra, to the need to use both \( \sigma_a \) and \( \sigma_b \) for presentation of \( \mu(f) \). Inhomogeneity of MS, as observed in [2], immediately appears as a peculiarity on \( \mu(f) \).

![Fig.4. Quantitative model for A2 PF](image)

(Fig.1; experimental spectrum for \( f > 10^8 \) Hz is extrapolated on for possibility to use KKR). Raman spectra for samples A2…A5 and B1…B5 (Fig. 2) show that MnZn-ferrites are more inverse than NiZn-ferrites.

**Table 1.** Data of NiZn and MnZn samples used in experimental proof of the modelling

<table>
<thead>
<tr>
<th>Group</th>
<th>№</th>
<th>Dimensions, mm</th>
<th>A, mm²</th>
<th>( \mu(0) )</th>
<th>( \mu(0)_{\text{exp}} )</th>
<th>( D, \mu m )</th>
<th>( \sigma_{nD} )</th>
<th>( \sigma_a )</th>
<th>( \sigma_b )</th>
<th>( \sigma_{av} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiZn</td>
<td>A1</td>
<td>R16x7x6</td>
<td>27</td>
<td>200</td>
<td>203</td>
<td>7.7</td>
<td>0.49</td>
<td>0.47</td>
<td>0.66</td>
<td>0.56</td>
</tr>
<tr>
<td></td>
<td>A2</td>
<td>R12x8x6</td>
<td>12</td>
<td>2000</td>
<td>2200</td>
<td>7.9</td>
<td>0.55</td>
<td>0.47</td>
<td>0.79</td>
<td>0.63</td>
</tr>
<tr>
<td></td>
<td>A3</td>
<td>R20x10x6</td>
<td>30</td>
<td>2000</td>
<td>2100</td>
<td>7.9</td>
<td>0.55</td>
<td>0.44</td>
<td>0.80</td>
<td>0.62</td>
</tr>
<tr>
<td></td>
<td>A4</td>
<td>R31.5x20.5x6</td>
<td>33</td>
<td>2000</td>
<td>2125</td>
<td>7.9</td>
<td>0.55</td>
<td>0.43</td>
<td>0.81</td>
<td>0.62</td>
</tr>
<tr>
<td></td>
<td>A5</td>
<td>R40x23.5x6</td>
<td>51</td>
<td>2000</td>
<td>2140</td>
<td>7.9</td>
<td>0.55</td>
<td>0.42</td>
<td>0.84</td>
<td>0.63</td>
</tr>
<tr>
<td>MnZn, 6000HM1</td>
<td>B1</td>
<td>R10x4.3x10</td>
<td>28.5</td>
<td>6000</td>
<td>6750</td>
<td>12.9</td>
<td>0.53</td>
<td>0.41</td>
<td>0.53</td>
<td>0.47</td>
</tr>
<tr>
<td></td>
<td>B2</td>
<td>R20x9.1x10</td>
<td>54.5</td>
<td>6000</td>
<td>6530</td>
<td>12.9</td>
<td>0.53</td>
<td>0.43</td>
<td>0.55</td>
<td>0.49</td>
</tr>
<tr>
<td></td>
<td>B3</td>
<td>R25x12x10</td>
<td>65</td>
<td>6000</td>
<td>6650</td>
<td>12.9</td>
<td>0.53</td>
<td>0.44</td>
<td>0.54</td>
<td>0.49</td>
</tr>
<tr>
<td></td>
<td>B4</td>
<td>R30x14.3x10</td>
<td>78.5</td>
<td>6000</td>
<td>6850</td>
<td>12.9</td>
<td>0.53</td>
<td>0.44</td>
<td>0.57</td>
<td>0.50</td>
</tr>
<tr>
<td></td>
<td>B5</td>
<td>R33x10x10</td>
<td>115</td>
<td>6000</td>
<td>6620</td>
<td>12.9</td>
<td>0.53</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

CIP spectra was measured up to adequately high frequencies to ensure all of ACp large amplitude dispersion region be included. Measurements were taken by R&S network analyzer ZVR-E, with attached specially designed measurement fixture with known parameters.

CMP spectrum (Fig.3) of medium-permeability NiZn ferrite with Co-additives (sample A1, Table 1) and of small size, measured some time ago [10], shows the correlation with its MS similar to now measured high-permeability ones of different sizes, but of similar MS (A2…A5; Fig. 4: practically
coinciding set of spectra). This similarity is achieved by the use of special process in the production of samples – by waterjet assisted mechanical cutting from single Ferroxcube 4S60 tile [11]. To relate the spectra with their MS (like as in [8]) averaged dispersion parameters $\sigma_{av} = (\sigma_a + \sigma_b)/2$ are given in Table 1; $\sigma_{av}$ shows that MS of the samples is rather homogenous but with considerable amount of defects within grains. In all cases approximations of experimental $\hat{\mu}(f)$ is excellent (Fig. 2) or good (Fig. 3).

In this context it is possible to state that situation for MnZn samples (as well cutted from single tile of 6000HM-1 ferrite [12]) depends on sizes: for rather small size cores (B1…B4, Fig. 5) picture is similar with already discussed NiZn samples; for cores of higher sizes (e.g., B5, Fig. 5) dimensional resonance [6] takes place – CMP ceases to have correlation with its MS, approximations by Eq. 1 are not physically meaningful.

4. Conclusion. A correlation made between the theoretical model of $\hat{\mu}(f)$ (based on account of effects coming into being from realities of actual MS of PF) and the experimental data evidenced that the mode is well suited for presentation of CMP as intrinsic characteristic of PF. The parameters needed for the presentation are clear physically and well understood in relation to typical, homogeneous MS of PF; as such the model may serve for prediction of magnetic spectra as well. In the full measure these statements corresponds to NiZn-ferrites; in the case of MnZn-ferrites the size of magnetic core exerts some effect on $\hat{\mu}(f)$: for small sizes there are no influence (the measured $\hat{\mu}(f)$ corresponds to intrinsic properties of PF); for bigger ones $\hat{\mu}(f)$ gradually turns into extrinsic characteristic which eventually identifies ferrite as resonator (exhibiting dimensional resonance).

Literature references
[12] www.rusgates.ru, 6000HM-1 material (in Russian)