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"Research Note"

EFFECT OF MoO_3 ADDITION ON THE Q FACTOR, CURIE TEMPERATURE AND LINEAR SHRINKAGE OF Mn-Zn FERRITES[*]

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Abstract– In this work MoO_3 was added to the Mn-Zn ferrite with the basic composition of (52mol% of Fe_2O_3, 34mol% of MnO, 14mol% of ZnO) at concentrations of 0.08, 0.5, 1 and 5 wt%. The optimum sintering condition was determined based on maximum Q factor values. The microstructure of the sintered samples was studied using SEM. The magnetic properties were characterized by determining Q factor and Curie temperature. Results showed that the addition of MoO_3 increased Curie temperature and noticeably decreased linear shrinkage. The Q factor was maximized at a concentration of 0.5 wt% MoO_3 making this value the best.

Keywords– Soft magnetic materials, Mn-Zn ferrite, Curie temperature, Q factor, linear shrinkage

1. INTRODUCTION

Tab (Mn-Zn ferrites) is very important ferromagnetic ceramics, particularly for application at high frequencies. The main motivation for the use of Mn-Zn ferrites in transformer cores and inductors is their high permeability and low losses, combined with a good thermal stability of permeability [1-4]. The optimum microstructure for an Mn-Zn high frequency ferrite core is a fine, uniform microstructure with highly resistive grain boundaries and low porosity. Small amounts of additives can greatly affect the properties of ferrites [5-9]. The need to develop high permeability low loss materials for the telecommunication industry led to the use of various additives in Mn-Zn ferrites [10-12].

In this work the samples were sintered in different sintering conditions. The optimum sintering condition was determined based on maximum Q factor values. The samples were sintered at 1200°C for 2 hours in the presence of nitrogen and oxygen.

2. EXPERIMENTAL METHODS

a) Sample preparation

Tab (Ferrite powder) with the composition 52% Fe_2O_3, 34% MnO and 14% ZnO (mole%) was conventionally prepared. Proper amounts of the raw oxides Fe_2O_3, MnO and ZnO were wet mixed and homogenized in a planetary Teflon ball mill for two hours using water as the wetting agent. Particle size after this stage was around 2 microns. A SEM micrograph was used for particle size measurement.

The dry powder was mixed with 5 wt% glycerin as a binder, to facilitate compaction into E-shape and toroid samples. For each composition, two E shape samples were prepared. Samples were pressed at a pressure of $3\ {}^{\text{ton}}\!/_{\text{cm}^2}$.

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The pressed samples were then sintered in a controlled atmosphere furnace in the presence of oxygen and nitrogen. The sintering temperature and time were 1200°C and 2 hours, respectively, which were determined experimentally to produce a spinel structure with the highest permeability and Q factor. To do so, a chart for heating Mn-Zn ferrites with a constant oxidation degree was consulted [3]. Partial pressure of oxygen and nitrogen was controlled by controlling the flow of these gases during heating up, sintering and the cooling down stages.

b) Curie temperature measurement

Curie temperature was measured by a vibrating sample magnetometer measurement.

c) Quality factor measurement

Q factor was measured by an LCR meter with the ferrite samples in the circuit. The frequency of the circuit was varied, and at the resonant frequency f_o, Q was calculated to be maximum.

d) Linear shrinkage measurement

Linear shrinkage was determined by measuring the maximum length of the E-shape sample after cold pressing and before sintering (l_0) and that of the specimen after sintering (l). The linear shrinkage was calculated by the following formula:

$$l.sh. = \frac{l - l_0}{l_0}\%$$

3. RESULTS AND DISCUSSION

a) Curie temperature

Table 1 shows that increasing the amount of MoO_3 to the Mn-Zn ferrite increased the Curie temperature. Unlike Zn, with a larger ionic radius compared to Mo($r_{Mo^{6+}} = 0.62Å$, in comparison to $r_{Zn^{2+}} = 0.74Å$) which reduces the Curie temperature by increasing the interatomic distance between magnetic sites [5], Mo increases this temperature. This observation can be explained by the following effect.

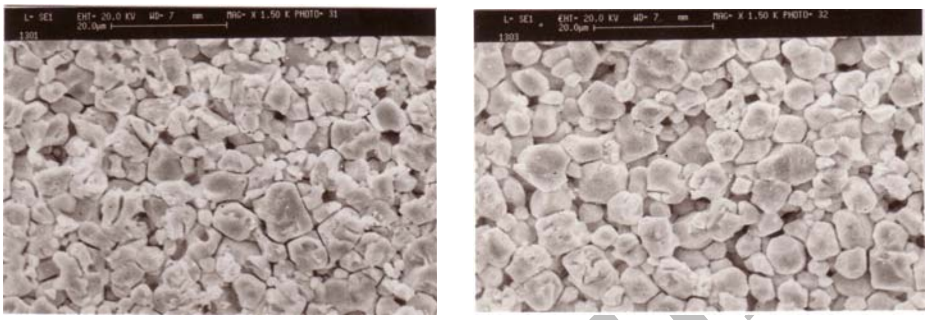
Table 1. Effect of MoO_3 addition on the Curie temperature

MoO_3 wt%	0	0.08	0.5	1	5
T_c°C	230	237	265	350	370

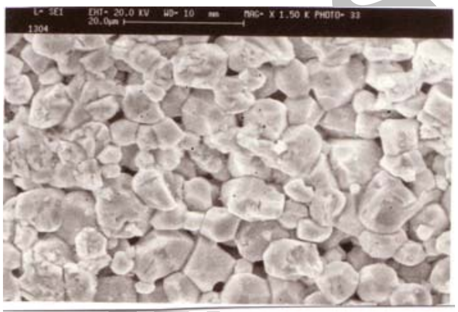
The Curie temperature separates the disordered paramagnetic phase from the ordered ferromagnetic phase. This depends on the numbers and types of paramagnetic ions which occupy A and/or B sites in spinel, causing different effects of C_A and C_B on the Curie temperature. When MoO_3 was added, because of the smaller ionic radius of Mo compared to Zn, Mo cations can easily diffuse into the lattice or magnetic sites. Also Mo^{6+} cations are strong positive centers. Therefore it will affect the Curie constants and T_C will be altered. Another view point is that due to the smaller ionic radius of Mo, it cannot reduce the Curie temperature by increasing the interatomic distance between magnetic sites, and a higher energy is needed to eliminate the ferrimagnetic order.

b) Q factor

Figure 1 shows the microstructure of three types of samples with different concentrations of MoO_3. Figure 2 shows the effect of the MoO_3 addition on the Q factor at a constant frequency (f=38 kHz). This oxide up to a 0.5wt% increased the Q factor. A decrease in Q was observed at a high concentration. With increasing MoO_3 up to a 0.5wt%, a fine and uniform microstructure, low porosity and shorter pore-pore distance were produced. But a higher amount of MoO_3 (>0.5wt %) decreased the Q factor. Since Mo cations have a small ionic radius and high mobility, these cations can easily diffuse in the spinel crystal structure and magnetic sites (A and B). Mo^{6+}, unlike Zn^{2+}, cannot increase the total magnetic moment of the lattice, so it decreases the magnetic moment of spinel structure and the energy loss in each cycle increases. For this reason, Q factor decreases.



a: 0.08 wt%MoO_3 b: 0.5wt% MoO_3



c: 5wt% MoO_3

Fig. 1. SEM micrographs of three types of samples with different amounts of MoO_3 addition

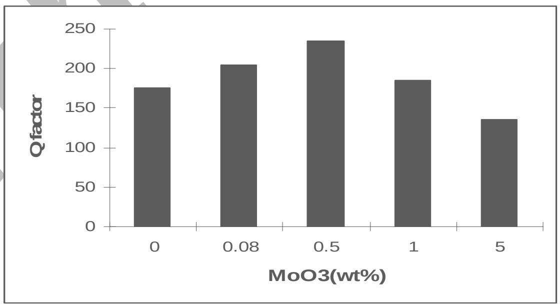


Fig. 2. Effect of MoO_3 addition on the Q factor

c) Linear shrinkage

Figure 3 shows a decrease in the linear shrinkage with increasing the amount of MoO_3. The shrinkage is strongly dependent on the amount of MoO_3 present in the samples. This behavior can be explained with the thermal behavior of MoO_3 which melts at 755°C and evaporates at 1195 °C [4]. At temperatures below 1195 °C, fast grain growth accompanied by pore coalescence dominates a rapid densification. In this stage MoO_3 is expected to stay as a liquid phase around the grain boundaries [4].

At the sintering temperature of this work 1200°C, MoO_3 is in the vapor state, but this temperature is not enough for MoO_3 to leave the matrix completely and MoO_3 with a high vapor pressure remains in the matrix, which prohibits material flow into the pores and densification will be stopped. This was shown experimentally, densification decreased with the increase in the amount of the % MoO_3.

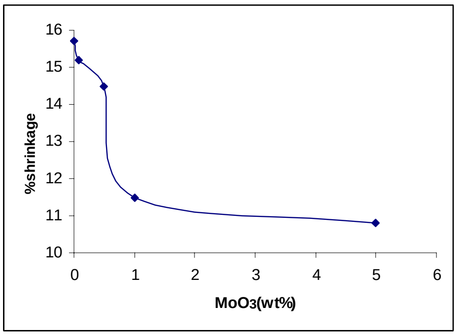


Fig. 3. Effect of MoO_3 addition on the linear shrinkage

4. CONCLUSIONS

It was concluded that the addition of MoO_3 to Mn-Zn ferrites (52mol% Fe_2O_3, 34mol% MnO, 14mol% ZnO) due to the small ionic radius of Mo^{6+} (o.62 Å), high electric positive charge (6+)of Mo^{6+} (o.62 Å) and also low melting point and high vapor pressure of the MoO_3 :

1- Curie temperature increases with increasing the MoO_3 concentration.
2- The linear shrinkage decreases by increasing the amount of MoO_3.
3- Q factor is maximum in the 0.5wt% of MoO_3 concentration and a decrease in Q factor was observed at high concentrations.

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