The influence of filler dispersity on radio-absorbing properties of material based on synthetic rubber

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Abstract. The article considers the features of changes in the radio-absorbing properties of an elastic absorber with a change in the dispersion of a functional filler. The technology of preparing the absorber and filling the covers of amplifiers in the microwave range is described. The results of the analysis of carbonyl iron powders by the methods of X-ray phase analysis, laser diffraction, scanning electron microscopy are presented. It has been shown that a decrease in the particle size of carbonyl iron by mechanical activation helps to reduce the frequency unevenness.

1. Introduction

The development of electronic equipment is associated with the development of materials that absorb electromagnetic radiation (EMR). They help to solve such problems as information protection, reducing radar visibility, increasing the directivity of antennas, as well as ensuring electromagnetic compatibility of electronic equipment [1, 2].

The relevance of this study is due to the creation of new radio-absorbing materials (RAM) with improved properties that can be used to absorb spurious emissions propagating in the form of electromagnetic waves in free space or in the form of surface waves along the surface of SHF-microunits.

In the production of microelectronic devices (MEDs), an elastic absorbing composition based on a polymer matrix is used, in which carbonyl iron powder is used as a filler. An analysis of the scientific and periodic literature and patents showed that the most promising and technologically advanced method for increasing the radio-absorbing properties of RAMs under production conditions is to change the dispersion of the functional filler of RAMs [3-5].

A known technology for producing powders of a functional filler of various dispersion by mechanical grinding in a laboratory bead mill in butanol medium with the addition of a surfactant, and the effect of the dispersion of the filler on the radio-absorbing properties of the composite has been studied [3]. It was shown that RAM filled with carbonyl iron powder after grinding has a reflection coefficient of no higher than -15 dB in the frequency range from 7.5 to 12.5 GHz, while a sample filled with powder in the initial state has reflection coefficients close to -1 dB, with a resonance at a frequency of 11 GHz.

In this regard, to increase the dispersion of the filler under production conditions, it is advisable to use the method of mechanical activation [6]. However, these questions in the literature were not discussed.

In connection with the foregoing, the aim of this work was to assess the possibility of increasing the radio-absorbing properties of the composite by increasing the dispersion of the filler as a result of its mechanical activation.
2. Objects and research methods

The object of the study was an elastic absorbing composition. The Vixint PK-68 compound - a low molecular weight synthetic rubber of the SKTN brand and cold vulcanization catalyst No. 68, was used as a polymer binder [7-9]. A filler is a powder of carbonyl iron of the P-10 grade with a particle size of 2 to 15 microns [10].

Mechanical activation (MA) of carbonyl iron was carried out in an AGO-2 water-cooled high-energy planetary mill («Novits Ltd»).

Sample 1. The original carbonyl iron.

Sample 2. Mechanically activated carbonyl iron. Ceramic balls with a diameter of 2 mm were used as grinding bodies, the ratio of the mass of the sample and grinding media was 1:40, the acceleration of grinding media was 30 g, and the MA time was 30 min.

Then experimental samples of elastic absorbent composition were made. Samples of carbonyl iron 1 and 2 were dried at a temperature of (120 ± 10) °C for 1-2 hours in an oven. SKTN low molecular weight synthetic rubber and carbonyl iron samples 1 and 2 were thoroughly mixed manually and heated for 0.5 h at a temperature of (120 ± 10) °C. Then the mixture was evacuated, a measured amount of catalyst No. 68 was introduced.

Thus, samples of the absorbing composition were prepared with carbonyl iron powder of different dispersion. The resulting absorbent compositions were poured directly into the slots of the MEDs covers. The polymerization duration is 24 hours at a temperature of (25 ± 10) °C. After polymerization, measurements were made of the frequency non-uniformity of amplifiers with covers of different compositions.

The phase composition of the samples was studied by X-ray phase analysis on a D8 Advance (Bruker) instrument in monochromatized CuKα radiation with a wavelength of 1.5418 Å. The voltage and glow current were 40 kV and 40 mA, respectively, scan step 2θ = 0.02 °, signal accumulation time 1 sec / dot, scan range 10-80 ° 2θ.

The particle size of the initial carbonyl iron powder and sample 2 after mechanical activation was determined by laser particle diffraction using a SALD-2101 device («Shimadzu»). A study was also conducted of the structure and surface morphology of the initial and mechanically activated carbonyl iron sample. The study was performed on a scanning electron microscope JSM-6610LV, «JEOL» with the attachment X-ray microanalysis INCAx-Act, «Oxford Instruments».

The absorption properties of RPMs were evaluated by the frequency non-uniformity of the amplifier, which was determined using a P2M-18 transmission and reflection coefficient modulus meter. This is one of the important characteristics of the amplifier, which shows the dependence of the power gain on the frequency of the signal. The elastic composition with which the covers of the amplifier are filled in, absorbs radiation and removes resonances, thereby improving the parameters of the amplifier. A decrease in the frequency non-uniformity of the amplifier will indicate an improvement in the radio-absorbing properties of the composite material.

3. Results and discussion

As a result of the experimental work on the effect of dispersion of the magnetic filler, the following results were obtained.

On the X-ray diffraction patterns (Figure 1) of the carbonyl iron powder in the initial state and in sample 2, reflections are observed, the broadening of which during grinding increases insignificantly, unlike the reflections on diffractograms in [3]. The difference is probably due to the fact that in this work we used dry mechanical activation of carbonyl iron powder, and in [3] - mechanical activation using surfactants as a stabilizing additive.

The particle size distribution of the studied carbonyl iron samples is shown in Figure 2.
Figure 1. X-ray diffractogram:
1 – the initial carbonyl iron powder; 2 – carbonyl iron powder after MA

Figure 2. Particle size distribution determined by laser diffraction.
Sample 1 – the initial carbonyl iron powder; sample 2 – carbonyl iron powder after MA
Based on the data of laser diffraction, it can be seen that sample 2 is a monodisperse system with particle sizes from 2 to 10 μm, in which up to 85% of the particles have a diameter of 4 μm. The initial sample is a polydisperse system with particle diameters from 2 to 15 μm, where the maximum proportion of particles with a diameter of 4 μm barely reaches 50%. Thus, mechanical activation increases the dispersion of powders by 1.7 times.

The structures of the particles of carbonyl iron powder and mechanically activated sample 2 are shown in Figure 3.

Based on the data of scanning electron microscopy, it was found that the particles of the initial carbonyl iron powder have a globular shape with particle diameters mostly about 10 μm. In the MA structure of carbonyl iron, after grinding with balls (sample 2), the formation of a new fraction of carbonyl iron powder with globular particles and sizes mostly 4 μm was observed.

As a result of measurements of the frequency non-uniformity of amplifiers with covers of different RAMs compositions, it was found that the amplifier with a cover filled with absorbing compound II (ΔK = 1.13 dB) has the minimum frequency non-uniformity, while the frequency non-uniformities ΔK of an amplifier with a cover without using an absorbing composition and an amplifier with a lid filled with absorbing compound I is 1.29 and 1.18 dB, respectively (Table 1).

**Table 1.** Frequency non-uniformity (ΔK, dB) of an amplifier with covers filled with different compositions.

| Composition number | RAM Fillers                  | Frequency non-uniformity ΔK, dB |
|--------------------|------------------------------|---------------------------------|
| -                  | without absorbent composition| 1.29                            |
| I                  | initial carbonyl iron - sample 1 | 1.18                           |
| II                 | MA - sample 2                | 1.13                            |
Thus, amplifier covers filled with RAM filled with mechanically activated carbonyl iron powder improve the parameters of the amplifier compared to covers filled with material filled with carbonyl iron starting powder by 5%, which reduces the amplifier tuning time.

Therefore, an increase in the dispersion of magnetic filler particles by mechanical activation helps to reduce the frequency non-uniformity and increase the radio-absorbing properties.

4. Conclusion

As a result of the research work, a method for increasing the radio-absorbing properties of RAMs by increasing the dispersion of a functional filler was tested.

It was found that mechanical activation increases the dispersion of carbonyl iron particles by 1.7 times, which reduces frequency non-uniformity by 5%.

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