# SYNTHESIS AND PROCESSING OF SOFT MAGNETIC THERMOPLAST NANO@MICROPARTICLE COMPOUNDS

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## Abstract
*In this work Nano@Microparticles were manufactured in small quantity by using reactive silanes with functional groups, which react by addition reaction with the surface of the nano and micro particles. Ceramic Microspheres W-210 (by 3M™) were used as the micro particles and the spherical Iron Oxide ($Fe_3O_4$, 98%, 20–40nm) nano particles were obtained from the company Nanostructured & Amorphous Materials Inc. The functionalization of the particles occurs stepwise under nitrogen atmosphere. The functionalized particles were investigated by FTIR measurement and compared with untreated micro particles.*

## 1 Introduction
The market for electronic devices demands an increased application of soft magnetic metal-plastic compounds (PBSMC), for example as a magnetic core for relays. For these applications PBSMCs can be produced by using micro injection moulding. The advantages of such advanced materials are: minimizing the present eddy current losses, allowing for an economical fabrication of high performance soft magnetic parts and achieving complex geometries.
The purpose of this work, which is based on the research of Bosse [1] and Kirchberg [2], is the functionalization of ceramic micro particles with magnetite ($Fe_3O_4$) nano particles by using reactive silanes with reactive groups and to couple them in an addition reaction to Nano@Microparticles. This coupling mechanism is based on the works of Ming et al. [3] and Viel [4]. The silica nano and micro particles were coupled by using reactive silanes with functional amino and epoxy groups. These groups react in addition reaction to raspberry-like particles as illustrated in Figure 1.
There are other variations for the functionalization of silica particles to enhance the adhesion between the polymer matrix und particle surfaces for increasing the mechanical properties of nano material composites [5]. However, these variations were not considered in this work. The particles were treated with different silanes with defined alkyl chain lengths and the interaction between the embedded particles and the polymer matrix were investigated.

For grafting polymers on a particle surface it is necessary to replace the Si-OH groups on the surface with more reactive Si-Cl groups, which allow the grafting of polymers on the particle surface [6].

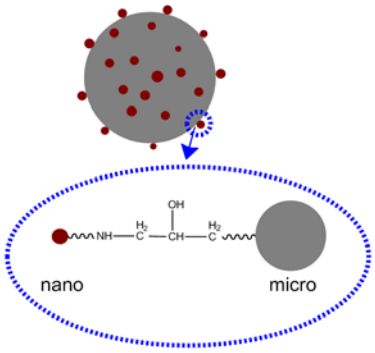


**Figure 1.** Modification of the micro particles with 3-glycidoxypropyltrimethoxysilane and of the nano particles with 3-aminopropyltrimethoxysilane; synthesis of the raspberry-like particles by addition reaction [3,4]

Soft magnetic materials significantly influence the characteristic functional properties such as magnetism, electrical and thermal conductivity, molding accuracy. Furthermore they have an influence on rheology and process parameters. The modification of the micro particles by coating them with a thin layer of nano particles is attempted in order to improve the degree of filling in the polymer matrix and to combine the super paramagnetic properties of the nano particles with the better dispersion properties of the micro particles. The resulting raspberry-shaped micro particles exhibit new material characteristics.

## 2 Materials and testing methods

For these experiments magnetite nano particles with a cubic crystalline structure produced by the company Nanostructured & Amorphous Materials Inc. were used. These particles are illustrated in figure 2a. The nano particles have a mean particle size of 30 nm (figure 2b). The particle size distribution was obtained by analysis of the TEM images by measuring the particle diameter.

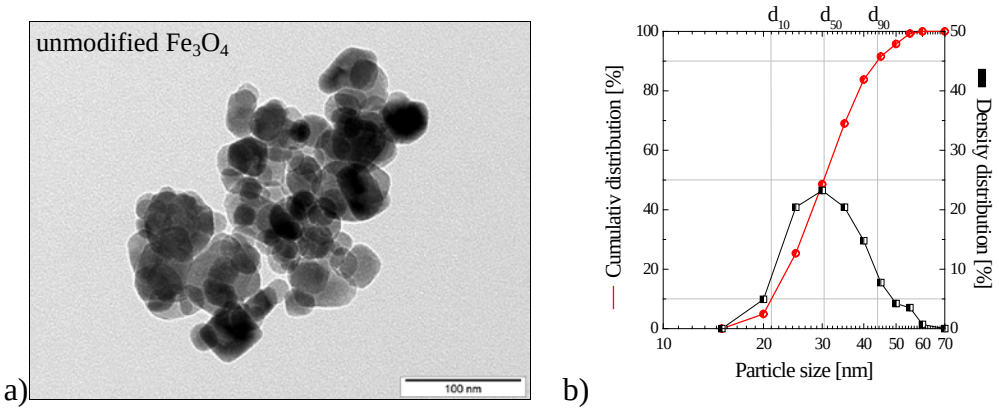


**Figure 2.** a) TEM image of the unmodified magnetite ($Fe_3O_4$) nano particles and b) particle size distribution

The ceramic micro particles, as shown in figure 3a, were obtained from the company 3M™. These partially crystalline particles are spherical. They have three distinct crystalline phases of potassium-, sodium-feldspar, quartz and one amorphous phase. The mean size was determined to be 4.3 µm, measured by laser granulometry (figure 3b).

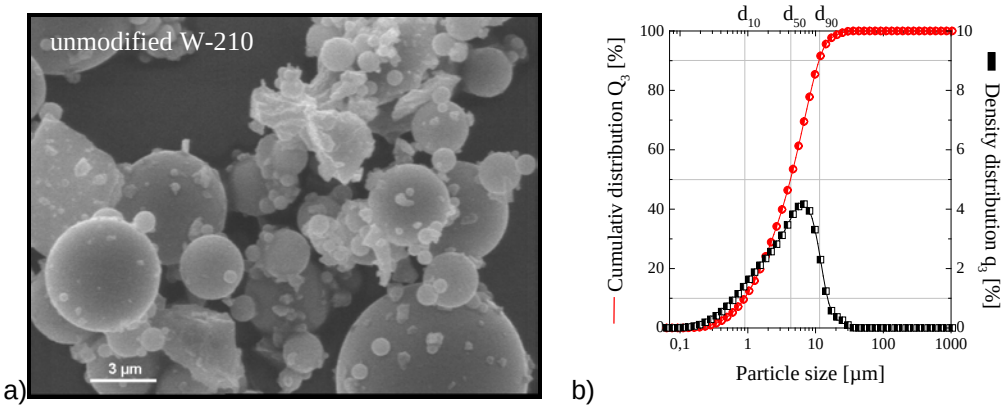


**Figure 3.** a) SEM overview of the unmodified ceramic (Microsheres W-210) micro particles and b) particle distribution with a mean particle size about 4,3 µm obtained by laser granulometry.

The two types of particles were treated with reactive silanes and coupled in the last step to Nano@Microparticles. The modification has to be done stepwise. In the first step 1 g of each particle type is separately dispersed in isopropanol. After this a few drops of chlorodimethylsilane were added to the dispersions and kept under nitrogen atmosphere at a temperature of 50 °C for two hours. In the next step the particles were modified with two different silanes with functional groups. For the modification of the nano particles 3-aminopropyltrimethoxysilane (APTMOS) was used at a temperature of 60 °C; for the micro particles 3-glycidoxypropyltrimethoxysilane (GPTMOS) at 50 °C was used. The nano particles were then treated under nitrogen atmosphere and stirred vigorously for twelve hours and the micro particles for 24 hours. After the treatment the particles were separated from the liquid by centrifugation and the supernatant was poured off. Subsequently the particles were dried in a vacuum oven at 50 °C for 16 hours. Finally, the particles were mixed together in a round bottom flask filled with isopropanol as solvent. They were let to react under vigorous stirring at a temperature of 75 °C for 24 hours.

## 3 Results

Qualitatively the FTIR measurement in figure 4 shows that there are interactions between the particle surface and the silanes. In this figure the unmodified particles are compared to the modified particles and the obtained Nano@Microparticles (green and purple line in figure 5). It can also be seen that nano particles are attached to the surface of the micro particles.

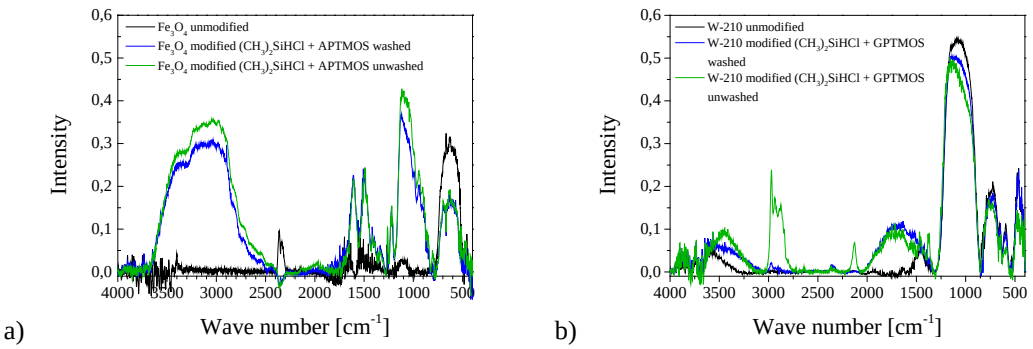


**Figure 4.** Qualitative comparison of the unmodified and modified nano a) and micro b) particles

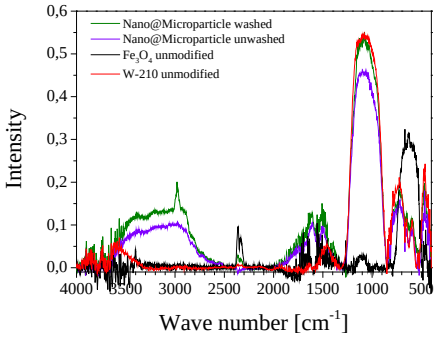


**Figure 5.** Qualitative comparison of the unmodified nano and micro particles and Nano@Microparticles

## 4 Conclusions

In this work it is shown that Nano@Microparticles can be synthesized with the described method. In future work the method for the synthesis of raspberry-like particles has to be optimized for manufacturing of larger quantities. Also it has to be shown by Atomic Force Microscopy (AFM) and X-ray Photoelectron Spectroscopy (XPS) that the surface of the micro particles is covered with modified nano particles. The particles have to be processed in a polymer matrix to soft magnetic plastic compounds and their characteristic functional properties have to be analyzed. Also the interaction between nano and micro particles as well as the Nano@Microparticles and the polymer matrix have to be investigated. Further research should focus on whether or not other types of particles can be coupled to Nano@Microparticles to obtain defined characteristic properties.

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