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Development of material based on nanostructured Cu-Nb alloy for high magnetic field coils of microsecond duration

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Abstract. The work is devoted to the development of a Cu-Nb composite material and an approach to the design of reliable tool coils, which require a magnetic field of about 40 T with a microsecond duration. A powder method has been applied to obtain homogeneous samples from a fine Cu-Nb composite alloy. The dependence of electrical and mechanical properties on annealing temperature was investigated. Layered sample was produced and tested under conditions of high magnetic field generation in comparison with a commercial wire.
Keywords: high magnetic field, inductor material, nanostructured Cu-Nb alloy, powder metallurgy.

1. Introduction

The problem of durability of materials used for the systems of multifold generation of high pulse magnetic fields (HMF) in the microsecond range of duration (half-wave duration less 100 μs) remains an urgent issue until now. Such magnetic fields are preferable in the technologies of magnetic pulse processing of metals: magnetic pulse welding (MPW), stamping, metal cutting, assembly operations, *etc.* [1–3], where the main instrument is a tool coil (inductor) in the form of a single or multi-turn coil. MPW is advanced technology which allows making solid-phase joining both similar and dissimilar materials [2] and hard-to-weld alloys, as well [3]. Poor durability of tool coils (inductors) for MPW is the main technical problem which constrains its worldwide application. As known, coil failure occurs at working surface where conductor is subjected to intense thermo-mechanical stresses under the force action of pulsed magnetic field and, especially, Joule heating [4]. To reduce the surface overheating of an inductor and, as a consequence, the thermal stresses, a conductor with inhomogeneous conductivity can be used [5]. At that, the conductivity may change monotonically or stepwisely. Theoretical description of inductor stressed state features under HMF can be found in our works for cylindrical [6] and flat geometries [7].

In the field of high-current electronic Cu-based nanostructured composites is of great interest due to the combination of high conductivity and abnormally high mechanical strength. The highest strength values are observed for pair Cu and Nb. Nanostructure is usually created from Cu-Nb alloy by the process of accumulative drawing and bundling (ADB) [8, 9]. Using nanostructured Cu-Nb wires lots of multiturn and/or multilayered inductor systems has been created for generating magnetic fields up to 75 T in subsecond range of duration [10–14]. The thermo-mechanical stresses can be decreased in such systems by increasing the number of turns which will lead to decreasing of current density in each one. However, high inductance of such systems makes it impossible to use it for generation of pulses in microsecond range of duration.

The aim of this work is to produce a material based on Cu-Nb alloy by powder approach, to study the effect of TiC addition on electrical, mechanical, structural properties and the behavior of the materials, including layered structures on their basis, under the generation of 40 T magnetic field of microsecond duration as compared to commercial microcomposite wire.

2. Experimental

2.1. Sample preparation

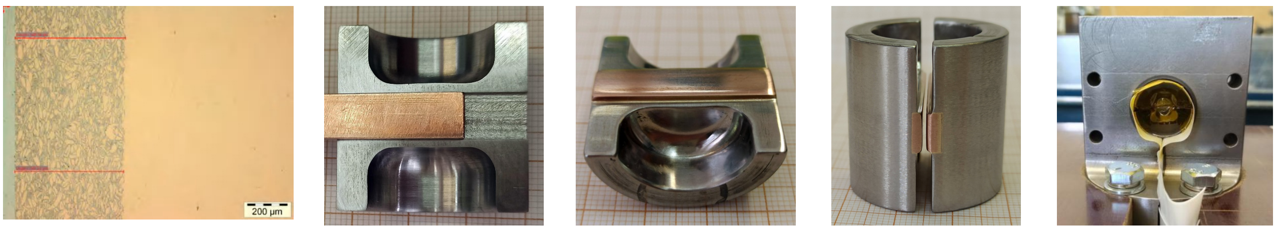
The process was started from Cu-18%Nb microcomposite wire of 0.18 mm in diameter (Nanoelectro, Russia). It was cut onto pieces of about 1 mm long, which were further disintegrated into base powder (BP) using planetary ball milling in petrol. The milled powder was sieved to size 20–64 μm; thus, the nanostructure of initial wire inside powder particles was retained. To form the layered structures with layers that differed in resistivity, TiC particles about 1.5 μm in size (Plasmotherm, Russia) were added to the base Cu-Nb powder (BP) in amount 15 vol.% through dry ball milling in argon for 15 min (denoted as BP15TC). As-milled powders were preannealed in vacuum at 500°C/1 h and then they were pressed into pellets up to 32 mm in dia by magnetic pulsed compaction (MPC) in evacuated to 10 Pa and preheated to 430°C mold at a pressure pulse of 1–1.3 GPa (for MPC see, *e.g.* [15]). After pressing the samples were tempered in vacuum at the same temperature for 2 h. Further annealing in vacuum (to 10^{-5} mbar) for 1 h at temperatures up to 850°C was performed to study its effect on material properties. Samples of commercial Cu-18%Nb multicore wire with cross-section (CS) 2×8 mm^2 of the same manufacturer were subjected to the same heat treatments.

2.2. Study of materials

The sintering kinetics and coefficient of thermal expansion (CTE) were studied in vacuum by dilatometry (DIL 402C, Netzsch) using the compacted cylinders of 8 mm in dia and long. Structural characteristics of materials were studied on the polished surface of disc samples before and after annealing using XRD (D8 Discover, Bruker AXS) in Bragg-Brentano-focused copper radiation with a graphite monochromator on the diffracted beam. Processing was performed using the TOPAS 3 software. Microstructure of the samples was studied by optical (Olympus BX41M) and atomic force (AFM Solver p47, NT MDT) microscopy both on polished and chemically etched surfaces. Etchants based on nitric acid as well as hydrochloric acid with ferric chloride were used for different samples. Vickers microhardness was studied using Nanotest 600 or Shimadzu DUH-211S, depending on sample type, at an indenter load 1 N and time exposure 5 s. The resistivity was determined on thin plates cut from 32 mm disk samples using 4-probe DC method; high precision LCR meter 6100 (GW Instek) equipped with a lab 4-point terminal block was used for resistance measurements. Total error for resistivity did not exceed 3–5%.

2.3. Durability test

To study the effect of high pulsed magnetic field generation on the inductor's surface degradation, the bars with a length of 32 mm and CS 2×8 mm^2, close to that of commercial wire, were cut from some compact samples of 32 mm in diameter, including the sample with bi-layer structure (Fig.1a).



(a) (b) (c) (d) (e)

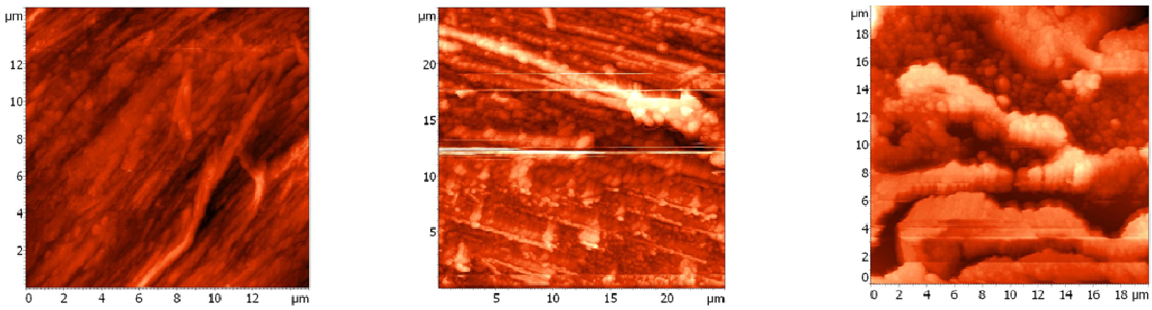
Fig.1. Layered sample cross section (a) and inductor system with MFC for durability tests (b-e).

It was done to compare the commercial and lab materials in close conditions. The bars cut of the pellets (commercial wire as reference material, too) were the brazed parts of a duplex magnetic flux concentrators (MFC) made of structural steel. Brazing the samples was performed using Ag-based alloy (40Ag-28.5Cd-17Zn-17Cu-0.5Ni-0.1Fe-0.05Pb-0.005Bi) at temperature around 750 °C. The assembled parts of the MFC in special insulation were then installed inside a capacitor-driven single-turn inductor (Fig.1b-e). Pulsed current generator PCG-135 (435 µF, 25 kV (max)) was used to energize the coil at a charging voltage about 7.4 kV (discharge current 570 kA). The magnetic field generated in the gap between the MFC parts was measured with a strip-type inductive sensor. The testing conditions were: peak magnetic field – 40 T, *T*/2 – 15 µs. The material brazed into the MFC is subjected to maximum thermomechanical stresses on the surface of its central part; optical imaging was used after each 5 pulses to study the evolution of the surface which was polished before the tests.

3. Results

3.1. Uniform samples properties

The BP and BP15TC powders were compacted by MPC to green densities about 97.3% and 93.7%, respectively. Analysis of the AFM image (Fig.2) of the etched surface of the BP sample indicates that the fibrous nanostructure is practically not preserved after high temperature annealing the compact. In the initial material (Cu-Nb wire), the niobium fibers are strictly ordered along the wire with individual fiber diameters not exceeding hundreds nanometers. During the milling process, the powder particles undergo plastic deformation in multiple directions, which probably results in disordered fibers. After pressing, the material is annealed at temperatures close to sintering ones. This is necessary to establish metallurgical bonds between the individual grains of the material, since the strength of the material is extremely important. On the other hand, heat treatment causes the niobium fibers to discontinue and coagulation into the spherical particles which was observed by the others [16, 17]. As annealed at 800°C, these niobium spherical particles have a diameter of 1 µm and greater with a tendency to agglomeration (it looks like that).



(a) (b) (c)

Fig.2. AFM images of etched surfaces of compact samples made of Cu-Nb wire pieces (a, b) and BP powder (c): (a) – after MPC, (b, c) – after MPC followed by 800°C/1 h annealing.

The processes of recrystallization and relaxation of the atomic lattices during annealing are confirmed by XRD analysis. Up to 800 °C the trends for Cu and Nb phases are the same: reduction of interplanar distance distortions, $\Delta d/d$, correlated with residual stresses and crystallites growth (Fig.3a). However, increasing the annealing temperature to 850°C leads to a violation of this trend, mainly for Nb phase. The most likely reasons are: dissolution of niobium atoms or oxygen into the copper matrix due to the milling process and recrystallisation of the second phases like Cu_2O, NbO and Nb_2C.

Fig.3b,c show the effect of annealing and TiC addition on the resistivity and microhardness of the samples made of BP and BP15TC powders as compared to the wire. The resistivity at the lowest annealing temperature was 2.6, 5.5 and 10.1 µOhm·cm, respectively. Annealing at temperatures up to 800°C led to monotonic decreasing the resistivity by about 18, 40 and 42%. At higher annealing temperatures the behavior slightly changes, especially for TiC doped material: an increase in resistivity was observed in this material. It’s probably due to the effect of the second phase, likely, orthorhombic Nb_2C (at such anneal), which content was twice higher in doped material than that in the base one. The addition of 15 vol.% TiC to the base material results in an almost twofold (1.8) increase in resistivity that is important for forming the layered structures. This ratio keeps constant after annealing at temperatures up to 800°C. The TiC addition also decreases the thermal expansion of the base material: CTE value was reduced by several percent, from about $19 \cdot 10^{-6}$ to $17 \cdot 10^{-6}$ K^{-1}.

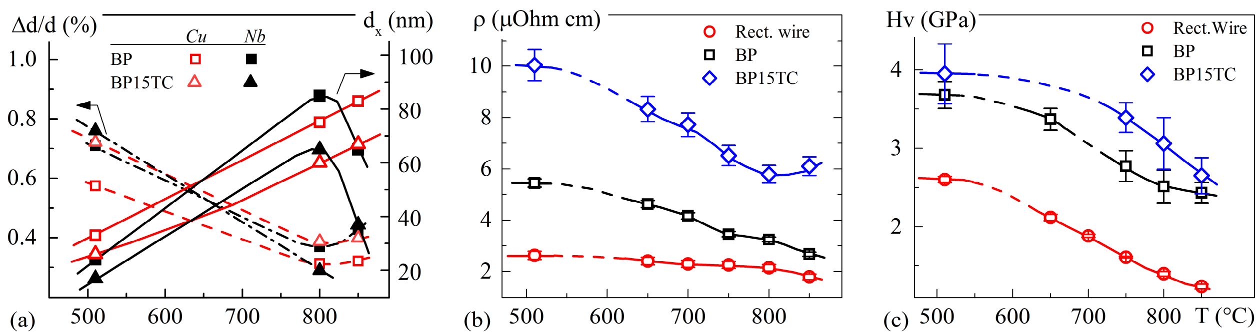


Fig.3. Effect of thermal annealing on structural characteristics (a, b), electrical resistivity (c) and microhardness Hv (d).

Similar temperature dependence was observed for the Hv microhardness (Fig.3c). The initial microhardness of the base powder sample was 1.5 times higher than that of the wire, which is due to the microstructure defects accumulated as a result of milling and pressing. The addition of TiC increases the hardness of the material from 3.5 to 3.9 GPa and provides the material with better thermal stability. The observed dependencies are in agreement with other studies on the thermal stability of fibrous nanostructured Cu-Nb composites [16, 18, 19].

3.2. Testing under HMF generation

For durability test, the double-layer sample was obtained using MPC followed by the vacuum sintering at 850 °C/1 h to provide it with more strength and better resistivity of support layer. In the initial state, high resistive layer had a thickness of 0.5 mm and a sharp interface with the support layer free of pores or cracks (Fig.1a). According to theoretical estimates, the thickness of resistive layer should be around 0.2 mm to achieve the proper effect. Therefore, it was thinned by grinding to a thickness of 0.3 mm after brazing the bar to the MFC.

MFC with brazed samples made of the commercial rect.wire and the double-layer structure were tested under conditions of HMF (Fig.1c–e). In this configuration, the ratio of the magnetic field in the gap between the MFC parts to the discharge current, B/I, was 71–73 T/MA depending on MFC configuration (at the same geometrical sizes of the brazed bars, this ratio depends on material resistivity and on a depth of magnetic field penetration as a consequence).

The behavior of the samples, as expected, varied. Optical images of surface for each sample are presented in Fig.4. The weak point of the wire is the pure copper spaced the Cu-Nb fiber composite cores. Each subsequent pulse resulted in residual plastic deformation, mainly, of copper interlayers and in morphological distinguishing of multicore structure of the wire. However, this effect is not critical. After 100 pulses of a 40 T field, the wire sample was still intact. From the change of surface color, we can say that there was a significant overheating led to material oxidation. In this study the lifetime of the wire was not limited by 120 HMF pulses of 40 T.

In this study, the two-layer structure exhibited no proper results. After first 10 pulses a brittle destruction of thin high-resistive layer occurred. The cracks were growing perpendicular to the current flow and reached the full thickness of the sample in 10–20 pulses. Perhaps it was thermal cracking at the microscale and further aggravation of cracks, which became visible across the current flow, due to the known saw-effect. Beneath the surface of the samples, it was transformed into cuts with splashes of metal coming out mainly in the direction of the magnetic field pressure: down below the plate plane and along the cracks from the plate. This fracture pattern may be caused to the combination of some effects connected with addition of TiC into the BP: it leads to a lower tensile strength and higher values of Hv. Also, it was probably worsened by choosing of suboptimal heat treatment. Nevertheless, this sample (structure) was able to withstand about 100 pulses of HMF of 40 T. Note, the results of testing the uniform sample made of base powder were done in [20].

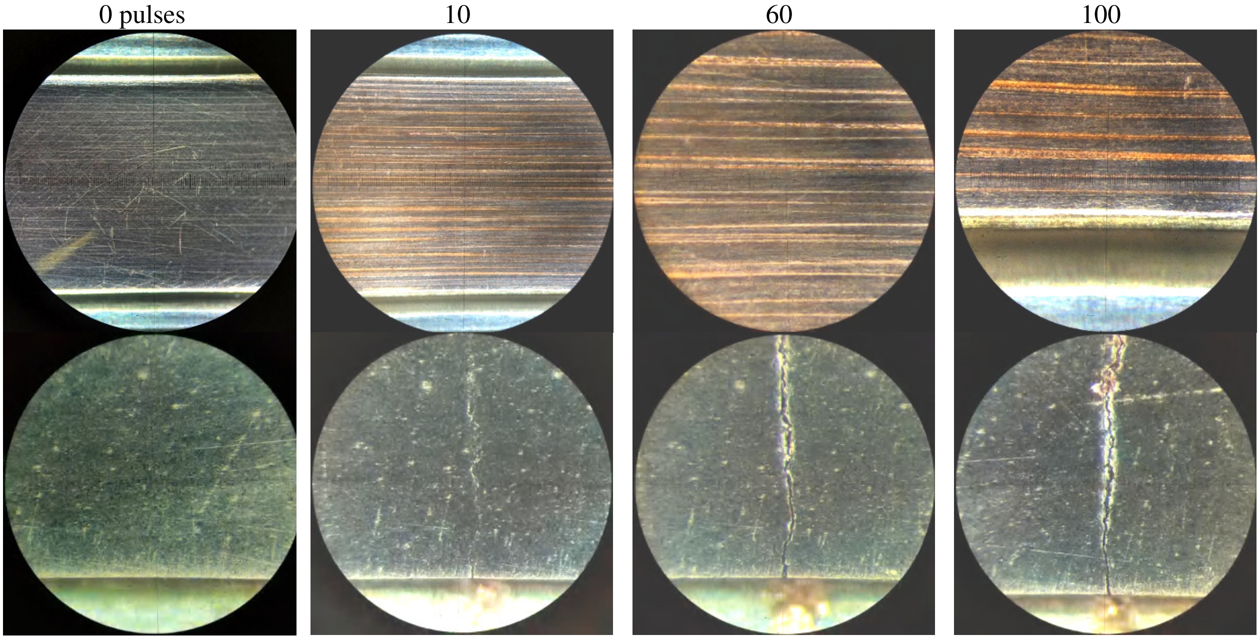


Fig.4. Surface evolution for rect. wire (top line) and layered sample (bottom line) during HMF tests.

4. Conclusion

Bulk uniform and two-layer samples based on Cu-Nb composites with relative density 94–97% were produced by magnetic pulsed compaction. Commercial fiber-like microcomposite Cu-18%Nb wire was used as raw material to prepare the press-powder by milling. To form the layered structures with layers differed in resistivity, 15 vol.% TiC particles were added to the base powder. Evolution of structural, electrical, and mechanical properties of composites with annealing temperature and the effect of TiC addition have been studied in comparison with the wire. The addition of 15% TiC to the base material results in an almost twofold increase in resistivity. This ratio keeps constant after annealing up to 800°C. The TiC addition also decreases the thermal expansion of the base material by several percent. The dependence of electrical resistivity and microhardness for both powder and reference samples (Cu-Nb wire) showed monotonical decrease after annealing from 500 to 750–800°C. At higher temperatures, 800–850°C, the properties of materials either change slowly or vice versa like for the TiC-doped material.

The two-layer sample with a substrate from the base material and 0.3 mm thick high-resistive surface layer with TiC addition was tested by generating a 40 T magnetic field (half-period 15 μs) as compared to commercial rectangular wire sample. The brittle fracture of the surface layer during the first 10–20 pulses was probably caused by an over-temperature annealing (sintering) process which could result in the formation of niobium or copper based second phases as well as tensile strength reduction rather than microhardness. Nevertheless, the double-layer structure was able to withstand about 100 pulses of HMF of 40 T. On the contrary, the lifetime of the rectangular wire (reference material) was not limited by 120 pulses of HMF of 40 T after that the wire sample was still intact. At such conditions the durability of Cu-Nb composite alloys is better than of beryllium bronze and they could be employed as inductor material for generating "fast" HMFs.

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