## SOLUTION COMBUSTION SYNTHESIS OF PURE METALL NANOPOWDERS

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Nanomaterials of a different nature have attracted significant research interest because of their unique size-dependent properties that are not typically observed in the corresponding bulk material. Nanomaterials are characterized by a high specific surface area to volume ratio, which is the basis of their novel physical-chemical properties [1, 2]. In this case, perhaps half or more atoms are near the surface or interface. This strongly affects as energy levels, electronic structure, and reactivity from the small structural and composition properties. Among the nanomaterials, metal nanoparticles (NP) are objects of a great research interest in modern materials chemistry and physics, where they find a wide application in such fields as nanoelectronics, optics, photochemistry, catalysis, etc [1-2].

Although, pure metals such as nickel, copper and cobalt was successfully produced by different combustion methods earlier [3-5], most of them are required an inert atmosphere ($N_2$, Ar) during synthesis process to prevent metal oxidation, or the using of addition post reductionof metal oxides in atmosphere of hydrogen. In spite of such kind of methods seems to be an attractive for metal NP production as an easier and more economically efficient there are only a few papers devoted to safe combustion one-step method of pure metal NP production in common atmosphere or without additional post reduction where glycine as a reducer was used.

Thus, in this work we report the synthesis of pure metallic Ni, Co and Cu NP by one-step modified SCS technique in normal air atmosphere without additional post reduction. The effect of the different fuels on the combustion synthesis process and structure of metal NP was also investigated.

*Synthesis procedure*. Metal NP (Ni, Co, Cu) samples were synthesized by modified SCS method using mixture of a metal precursor/oxidizer with a fuel. All chemicals were purchased from Sigma-Aldrich unless stated otherwise. Nickel nitrate hydrate ($Ni(NO_3)_2 \cdot 6H_2O$, Alfa Aesar, 98%), was used as a metal precursor, urea ($CH_4N_2O$, U, 98.6%), citric acid hydrate ($C_6H_8O_7 \cdot H_2O$, CA, 99%), glycine ($C_2H_5NO_2$, G, 98.5%) and hexamethylenetetramine ($C_6H_{12}N_4$, HMT, 99.2%) as a fuel component. For all experiments, the ratio (φ) between the fuel and the oxidizer was kept constant and equal to 2.Allsamples were obtained by the same procedure. Metal nitrate hydrate was dissolved in a minimum amount of distilled hot water (solution 1) and a fuel was dissolved in aqueous solution of ammonia (solution 2). Than solutions 1 and 2 were mixed and sol was formed. The obtained sol has been rapidly drying in microwave furnace, until gel and then foam has formed. The foam was ignited and burned at 873 K under normal air atmosphere, leading to the formation of a fluffy powder, which was rapidly cooled to prevent metal oxidation. A temperature change of the reaction mixture was measured using a K–type thermocouple. The output signal of the thermocouple was collected by a data acquisition system.

Figure 1 shows the XRD patterns of the synthesized Ni-, Co- and Cu-based materials in systems with different types of the fuel. It can be seen that the metallic phase was obtained in all cases.

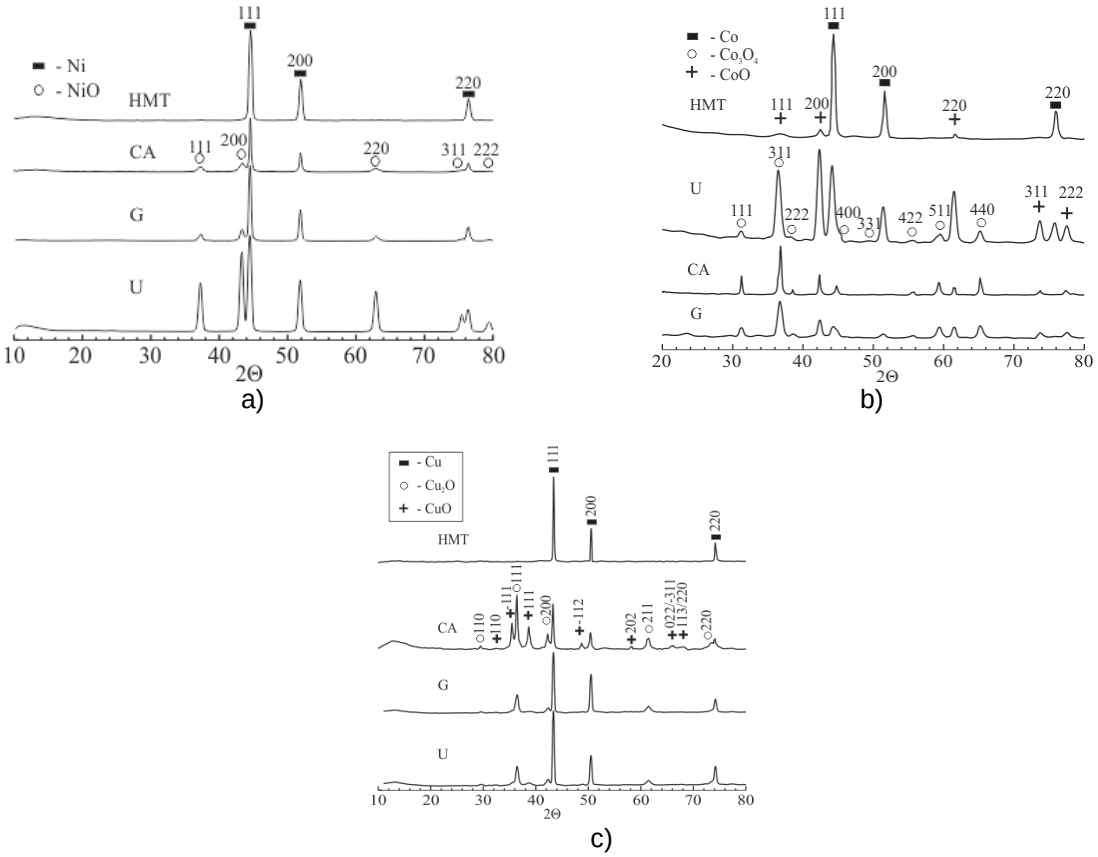


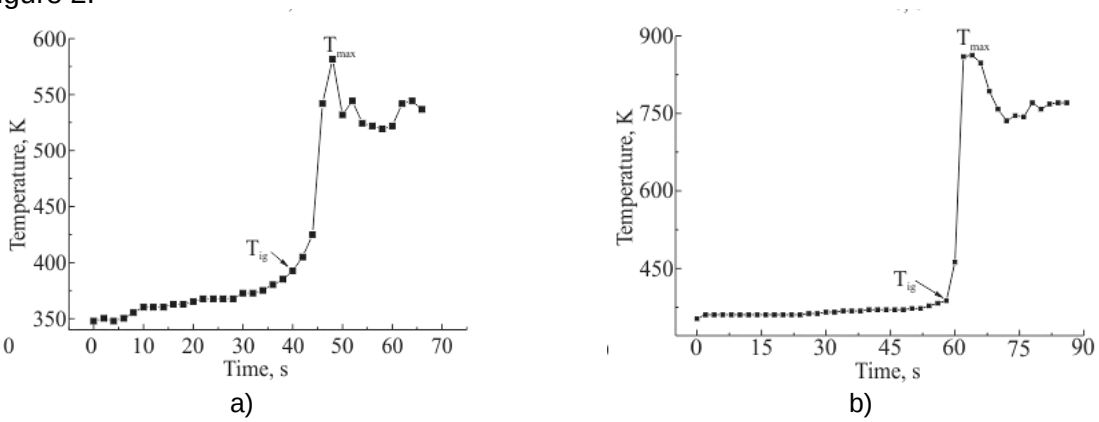

Figure 1 – XRD patterns of the synthesized Ni- (a), Co- (b) and Cu-based (c) materials

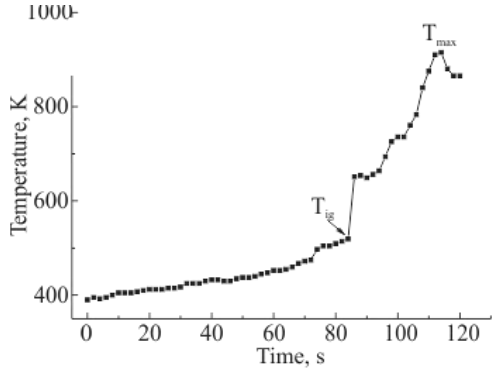
Nevertheless, only powder where HMT was used is characterized as a pure metallic Ni and Cu without any detected secondary phases and Co with a small amount of CoO as an additional side-phase.

In cases synthesis of Ni which CA and G were used traces of NiO phase were also detected. The usage of U as a fuel leads to formation of a mixture of Ni and NiO crystal phases. In cases where U and G were used $Cu_2O$ phase was also detected. The usage of CA as a fuel in processes of the synthesis of Cu leads to formation of a mixture of Cu, $Cu_2O$ and CuO crystal phases. In the cases synthesis of Co which CA and G were used a mixture of CoO and $Co_3O_4$ was detected as a main phases. The use of urea as a fuel leads to the formation of a mixture of Co, CoO and $Co_3O_4$crystal phases.

In the case of using of HMT fuel was observed formation of pure metallic with the nanoscale grain size Ni (~5–20 nm), Co (~5–40 nm) and Cu (~10-67 nm).

Experimental temperature-time profiles of SCS in $Me(NO_3)_2$ – HMT systems are shown in Figure 2.






c)

Figure 2 – Time-temperature-profile of SCS in $Me(NO_3)_2$–HMT systems, where Me – Ni (a), Co (b) and Cu (c)

In the system metal nitrate – HMT the one-step decomposition of HMT is resulted in formation of nitrogen and carbon monoxides and ammonia. The release of high amount of gases promotes the foam formation. After the accumulation of the gas mixture the exothermic reaction occurs at ($T_{ig}$) 393 K for nickel, 387 K for cobalt and 519 K for copper systems. Maximum temperature of combustion processes was about 570 K, 850 K and 940 K for nickel, cobalt and copper systems respectively. In all cases metal nitrate decomposition occurs during the synthesis process with the formation of oxide solid phase and nitrogen oxides. Then the reduction of oxide particles by gaseous $NH_3$, CO, etc. occurs.

**References:**

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