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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Fe/MgO MICRO-NANO COMPOSITE FOR ELECTROTECHNICAL APPLICATIONS

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Abstract

The composite based on the microns iron size powder and MgO nano-powder was prepared using pressing followed by conventional and microwave sintering. Microstructure of the composite was investigated to evaluate the changes induced by different sintering technology. Young's modulus, flexural strength and hardness of composites were analyzed to investigate the mechanical properties in dependence on MgO content, as well as in dependence on the sintering method. Microstructure and mechanical properties as well as functional magnetic properties of prepared composites are discussed in the paper. The main benefit of microwave heating found within process time shortening was confirmed in the case of the microwave sintered Fe/MgO composite.

Keywords: soft magnetic composites, microwave sintering, microstructural analysis, mechanical properties, functional properties

INTRODUCTION

L.J. Huang in his work [1] asks the question: "Microstructurally inhomogeneous composites: Is a homogeneous reinforcement distribution optimal?". A wide range of uniquely multi-scale structures have been successfully designed and fabricated by tailoring reinforcement distribution for discontinuous metal matrix composites in order to obtain superior performance, focusing on mechanical behaviour of the materials. There are a lot of functional composites, where electrical, magnetic or other physical properties are essential for application. Controlled microstructural inhomogeneity can help to improve the desired properties of functional composites. One of the functional materials is soft magnetic composites (SMC). The concept of SMC is based on the ferromagnetic powder particle surrounded by dielectric thin film [2]. Consolidation of this powder creates a metal matrix composite with a dielectric network. The high resistivity of the SMC is a way to achieve lower Eddy current losses. The main goal is to make these materials suitable in AC applications of middle or higher frequencies.

The fabrication of an as thin as possible dielectric layer and its preservation during powder consolidation is a key problem of these materials. Soft magnetic powders with ceramic secondary constituent are powder materials characterized by limited compressibility [3-4]. Compaction of these powders leads to low green density. The result of the specific sintering process, where an iron-iron connection is unwanted, is relatively high porosity of the final SMCs. High porosity means low permeability and magnetic flux density. Mechanical properties of SMCs are low due to the brittle network as well as high rate of porosity.

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Advanced powder metallurgy techniques can contribute to find solutions. One of the progressive sintering methods, which apart from process time shortening can help to preserve nano-structures in the sintering process, is microwave heating technology [5-6]. This work is focused on investigation of microstructure, electro-magnetic and mechanical properties of SMC based on pure iron micro-powder and high dielectric MgO nano-particles, prepared using traditional sintering as well as microwave sintering process.

EXPERIMENTAL MATERIALS AND METHODS

Technically pure Fe micro-particles (ASC 100.29, Höganäs AB, median size of powder particles $d_{0.5}$=100 μm) were dry coated by MgO nano-particles (MTI Corp., mean size d=30 nm, narrow distribution) using the Resonant Acoustic Mixing method in a Resodyn LabRAM mixer. Fe/MgO powders with 1, 2, 3, 5, 10 and 13.85 wt.% of MgO content were uniaxially cold pressed at a pressure of 600 MPa. Green compacts were sintered using a muffle furnace (conventional sintering further CS) at 600ºC, for 60 minutes, in a dry air atmosphere. Microwave sintering (further MWS) was provided in the multimode microwave cavity with controlled power from 300 W to 3 kW at a constant temperature of 600ºC for 15 minutes, in dry air atmosphere. The temperature in MW oven Hamilab V3000 was measured by IR pyrometer.

The hardness HV10 of cylinder samples with diameter size of 10 mm and height of 3 mm was tested by hardness tester LM 700AT. The elastic properties of prism bar sample size of 4x5x20 mm and ring samples` size of outer diameter 28 mm, inner diameter 25 mm and height of 3 mm were measured by non-destructive impulse excitation technique according ASTM E1876 using Buzz-o-Sonic equipment. Transverse rupture strength (TRS) was measured by 3 point bending test using universal testing machine LabTest 5.600 ZL. Light microscopy (LM Olympus GX71) and scanning electron microscopy (SEM JEOL JSM-7000F with EDS analyzer Inca) were used to investigate microstructure of the composite. Coercive force was measured by Coercive force meter DX-320 using a method of measurement of the coercivity of magnetic materials in an open magnetic circuit IEC 60404-7.

RESULTS AND DISCUSSION

Microstructure observation in Fig.1 and 2 show the network of secondary phase at the origin particle boundaries. Polarized light was used to study the distribution of secondary phase and porosity. Secondary phase was observed in the range of colours from light yellow to light grey in contrast to the dark brown iron matrix and black pores. Fe-MgO interphase of the sintered composites were investigated by SEM and line EDS methods. Low MgO content 1-2 wt.% is insufficient to avoid creation of iron-iron connections in both CS and MW sintering processes. Increased MgO content to 3 wt.% creates a continuous network of secondary phase. Increasing MgO content above 3 wt.% leads to the creation of thick interphase as well as to MgO cluster formation. Porosity depends on MgO content as well as on sintering technology. Pores are small and localised in three particle connection regions. In the case of CS composites, increasing MgO content increases porosity of the composites. Pores are larger in the composite with higher MgO content. Porosity was observed in interphase at higher MgO content in the composite. MW sintered composite is characterized by lower porosity in comparison to CS composite at the same MgO contents. A small addition of MgO content to 2 wt.% decreased the porosity of the composite. Interphase regions of MW composites are more compact with relatively more regular thickness in comparison to CS composites.

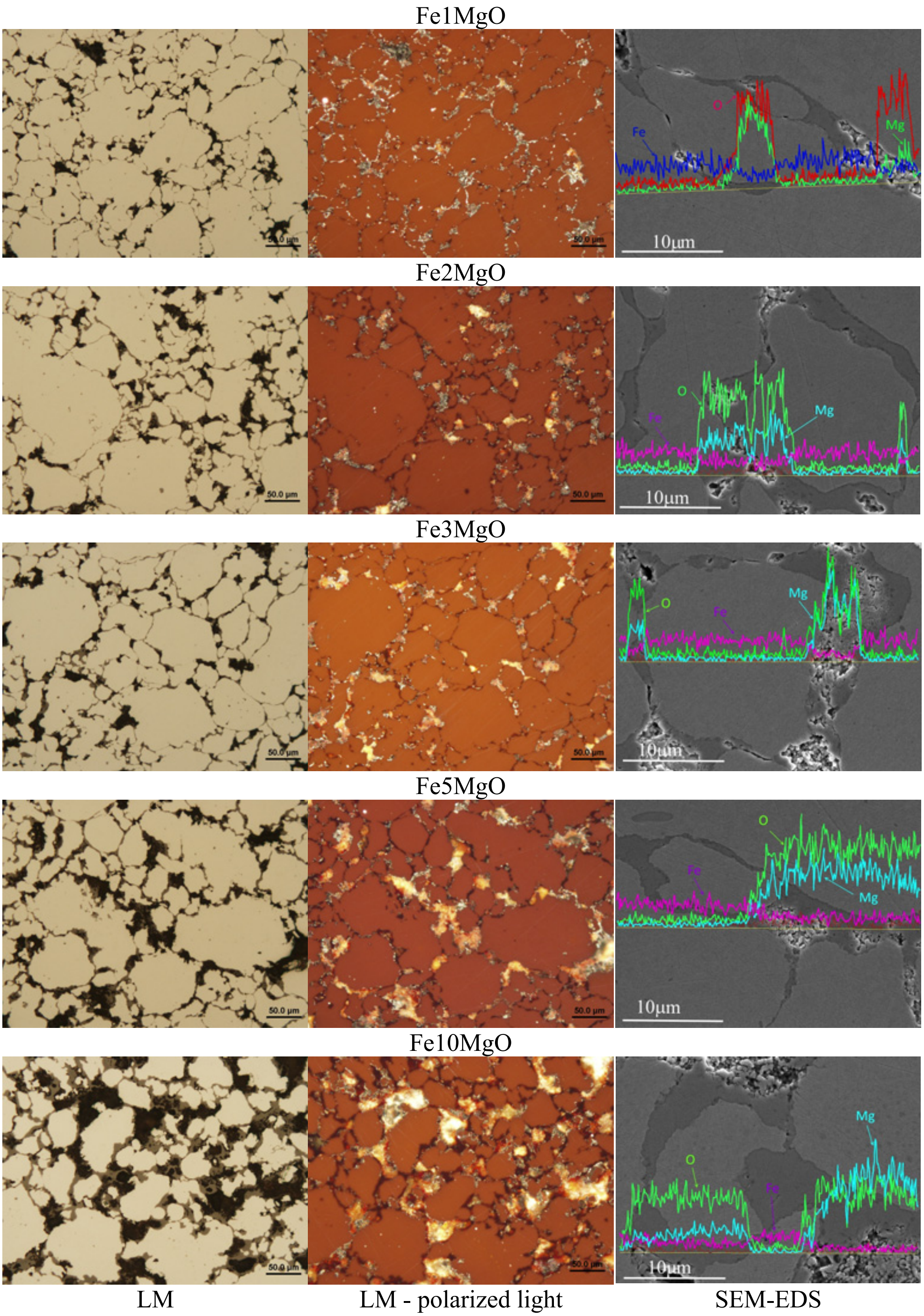
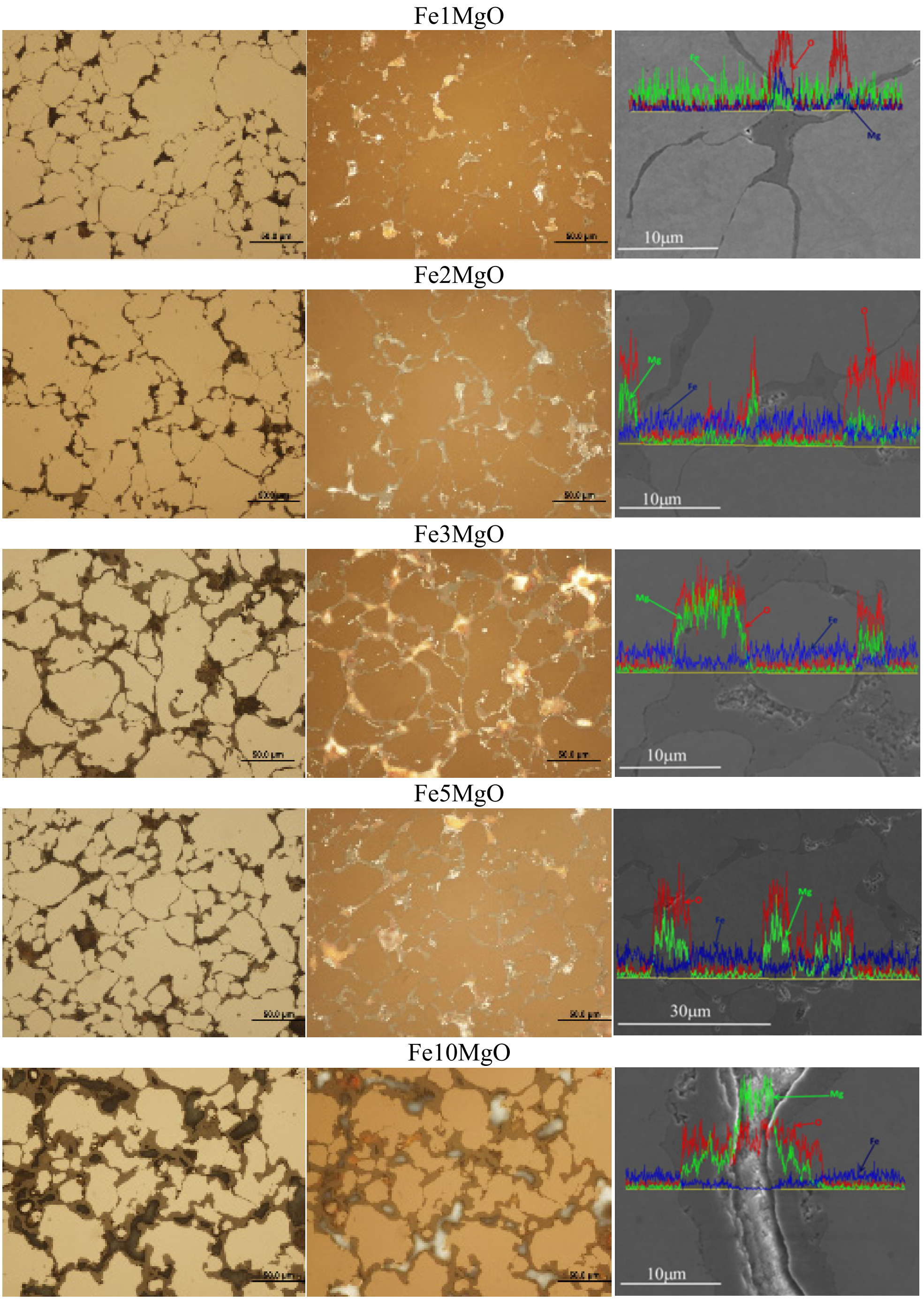
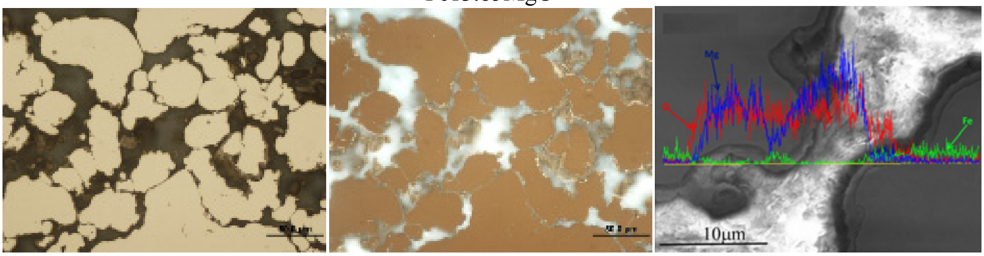


Fig.1. Microstructure and line EDS analysis of conventional sintered Fe/MgO composites.


Fe1MgO
10μm
Fe2MgO
10μm
Fe3MgO
10μm
Fe5MgO
30μm
Fe10MgO
10μm

Fe13.85MgO



LM LM - polarized light SEM-EDS

Fig.2. Microstructure and line EDS analysis of microwave sintered Fe/MgO composites.

Density values (in Fig.3.) confirm higher relative density of MWS composite in comparison with CS composite as well as in comparison with Fe/MgO green compacts. On the other hand, relative density values of the CS composites are lower than that of green compacts, because voids in the interphase decrease the density values especially at higher MgO content above 3 wt.%. The rise in value of Young's modulus after CS and MW sintering (in Fig.4.) confirms heat induced densification of MgO based ceramic phase in both sintering processes. Decreasing Young`s modulus in dependence on MgO content indicates increasing porosity values. Porosity has stronger influence on elastic properties than increasing content of MgO ceramic phase as it was calculated by Manoylov et al. [7].

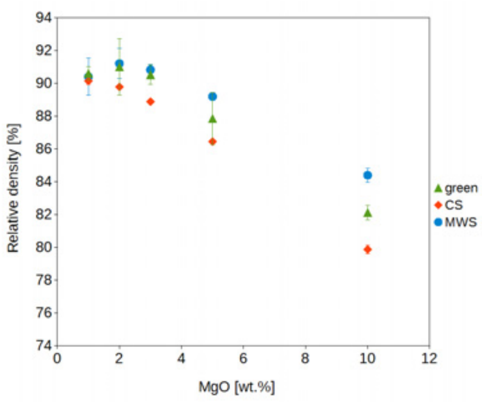


Fig.3. Relative density of the green compact, CS and MWS composites.

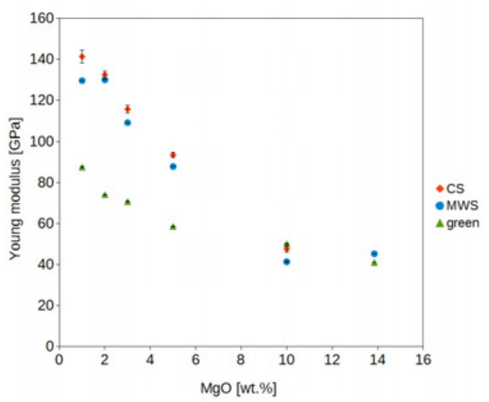


Fig.4. Young modulus of the green compact and sintered composites.

Analogous to Young`s modulus is the tendency of bending strength values in Fig.5. Strength represented by TRS value is related to the strength of secondary phase network. There are not Fe-Fe connections at higher MgO content, that is why TRS value decreases rapidly with an increased MgO content. Higher content of MgO means higher porosity, high porosity leads to lower elastic properties as well as lower mechanical strength. There is no significant difference in the TRS values of CS and MWS composites. More significant influence of sintering technology can be observed in the case of the hardness measurement. CS composite is characterized by two typical hardness values, which in Fig.6. represent Fe matrix (CS-1) and MgO based secondary phase (CS-2). Hardness values of the secondary phase of MWS composite (MWS-2) are considerably higher. The MW sintering process together with higher MgO content induces formation of a microstructure region characterized by low hardness value (MWS-3), while hardness value of the Fe matrix remains the same in both sintering processes. Mechanical properties of the soft magnetic composites are relatively low in comparison with sintered metals or structural composites. Functional properties are crucial to the application of SMC. However, mechanical properties have to fulfill minimal requirements which depend on the concrete type of application in electrotechnics. Functional properties of the SMCs are based on the balance of electric resistivity, magnetic permeability, coercivity and magnetic flux density. DC resistivity and coercive force measurement were used to investigate a basic electromagnetic properties development in dependence on MgO content and sintering technology.

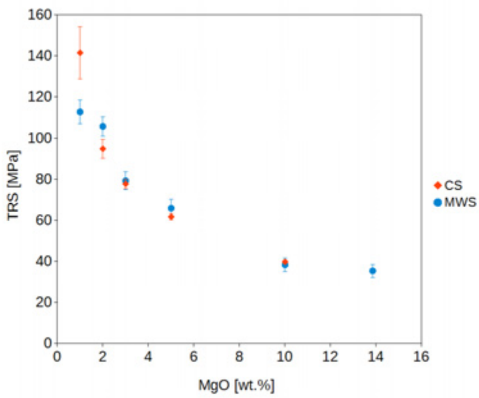


Fig.5. Transverse rupture strength of the sintered composites.

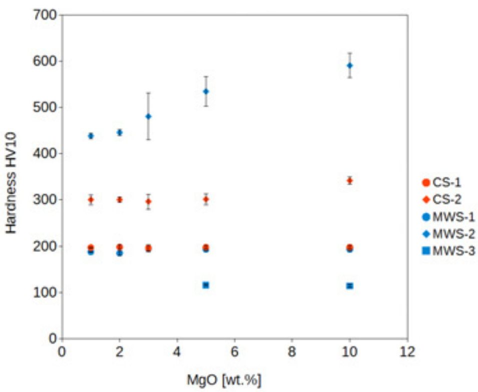


Fig.6. Hardness of the sintered composites.

Resistivity of the sintered composites is from 3 to 5 orders higher than that of green compacts as is shown in Fig.7. It was expected, because the result of the densification process during sintering is formation of Fe-Fe contacts to a certain extent. Resistivity of MWS composite Fe/3MgO is more then one order higher than that of Fe/2MgO, which confirms continuous dielectric network formation at MgO content above the value of 2 wt.%. In the case of conventional sintering it is necessary to use higher MgO content to create an effective dielectric network. Higher resistivity of CS composite at High MgO content (10 wt.%) is induced by higher porosity of CS in comparison to MWS composite. High resistivity created by high porosity is unwanted, because there can be expected a significant decrease of permeability in this case.

Coercivity as low as possible is required for soft magnetic composites. In Figure 8 it is shown that Fe/xMgO green compacts have coercivity force value circa 350 A/m. Coercivity of the green compact is influenced by cold pressing. Restoration and recrystallyzation processes during sintering are needed to decrease the coercivity value. Coercive forces decrease in CS composite in dependence on MgO content up to a value of 5 wt.%. In the case of MWS composite, coercive force decreases with increasing MgO content to a value of 3 wt.% MgO. Higher content of MgO above 3 wt.% or 5 wt.% leads to a rapid increase of coercive force of both CS and MWS composites. The most significant increase of coercivity can be observed in the case of MWS Fe/10MgO composite. Two factors can be responsible for the rise of coercive force values. The first factor is uncompensated mechanical stress in the sintered composite as a result of a different temperature expansion coefficient of primary and secondary phase. The second factor is additional phase formation in the sintering process. This additional phase can be non-stochiometric $MgFe_2O_4$ spinnel ferrite as it is shown in the study of Deraz etal. [8]. The cubic spinnel phases of $MgFe_2O_4$ are formed by microwave heating at 500ºC for low soaking time of 10 min as it was confirmed in [9]. More intensive magnesium ferrite spinnel formation can develop higher residual thermal stresses in MWS composite, which result is higher coercive force.

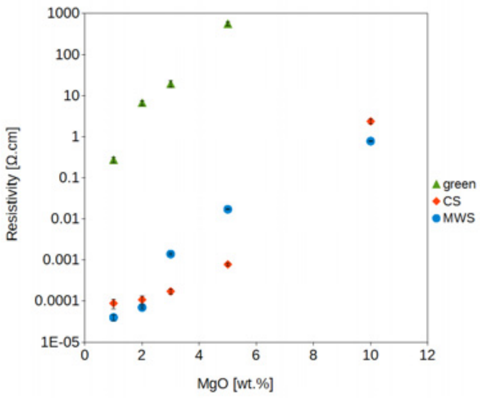


Fig.7. Resistivity of the green compact and sintered composites.

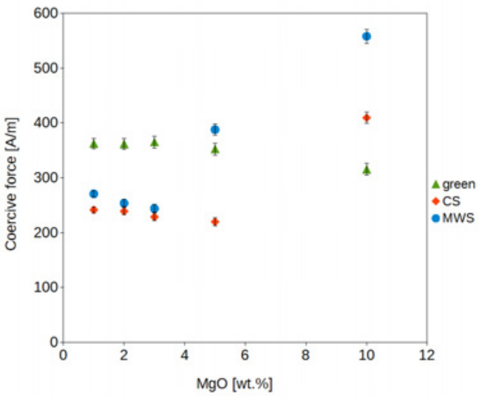


Fig.8. Coercive force of the green compact and sintered composites.

CONCLUSIONS

Soft magnetic micro-nano Fe/xMgO (x is from 1 to 13.85 wt.%) composite was prepared using cold pressing followed by traditional sintering as well as microwave sintering. Microstructure, mechanical and electro-magnetic properties were investigated to clear the processes of functional properties formation during sintering. Mechanical properties of Fe/MgO composite based on a lower content of MgO about 2 wt.% are comparable to current industrial soft magnetic composites. Higher contents of MgO above 3 wt.% MgO are typical of high resistivity and low coercive force values. The microwave sintering process helps to improve continuity of dielectric secondary phase network as well as contributes to a higher density of the composite, while process time is 5 times shorter in comparison to the conventional sintering process. An advantage of Fe/MgO soft magnetic composite is high temperature stability. Heat treatment at high temperature contributes to structure recovery, stress relief, thus give the possibility to optimize magnetic properties of the composite for specific applications.

Acknowledgements

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