Investigations on the strength of soldered joints of materials based on REM – Fe (Co) alloys

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Discovery and progressive development of the higher power permanent magnetic medium is the principle aspect of magnetism exploration. Producing magnets, composed of rare-earth metals, iron and boron (REM – Fe – B), marks an epoch in the permanent magnet investigations and technologies. An energetic product, which outperforms all characteristics of preceding magnetic mediums, has been achieved in the Nd – Fe – B-based magnets.

In contrast to alloys of the Nd – Fe – B system, the Sm – Co magnets combine high corrosion/ oxidation resistance and higher Curie temperature with high magnetocrystal anisotropy. The Sm – Co magnets perfectly suit for special usage in defense technologies and high-technology branches of production.

Strength of soldered joints of materials based on Nd – Fe – B and Sm – Co alloys is studied. Soldering of the materials has been carried out with the use of preliminary applied copper covering, deposited by cold spraying and galvanic method. The rupture strength of the samples soldered over copper covering, obtained by cold spraying, was demonstrated to be 1.8–2.3 times higher than that of the samples, soldered over electroplating.

Thermocycling of the samples within the temperature range from –50 to 150 ^{o}C and their corrosion tests have revealed that the soldered joints over copper covering, applied by cold spraying, are less tolerant to temperature differences, but have substantially higher corrosion stability, and at the same time their rupture strength is 1.5–2 times greater than that of the samples based on copper electroplating.

Rupture strength of the soldered joint for a 5 μm thick copper covering amounts to 48 MPa, which is 3–4 higher than that of the glued joints.

Key words: rare-earth alloys, magnetic mediums, soldering, strength of soldered joints, cold spraying, galvanic method, thermocycling.

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Introduction

Nowadays, materials based on REM – Fe (Co) alloys are widely used in different devices and instruments as permanent magnets (Pr – Fe – B, Nd – Fe – B, Sm – Co [1–6]), magnetostrictive elements (Dy – Tb – Fe [7–10]), etc.

On the other hand, usage of theses materials is accompanied by the problem of their reliable connection with one another as well as with the other structural materials (stainless and soft magnetic steels, non-ferrous metals, etc.).

Such bonding techniques as agglutination or mechanical attaching by special clamps or bands are the most commonly used to join the above mentioned materials. However, all of the listed means are not without disadvantages. The glued joints are of poor strength (8–12 MPa [11]). The mechanical methods are insufficiently effective because of low resistance to impact of the given materials and cracking susceptibility on contact mechanical stresses.

As regards the soldering, it is practically unused for connecting the given materials through their high activity and low corrosion stability. Besides, when using the soldering, there occur some problems of the flux or gaseous environment choice allowing to provide oxides removal from the surfaces of these materials and at the same time, excluding any possibility of the brittle REM-based intermetallides formation in the weld area.

As a matter of principle, all above mentioned problems can be solved by means of protective metal coats with high wettability to copper or nickel solders, preliminary applied on the surfaces of connectable materials [12].

However, using standard chemical or galvanic methods of applying that sort of coatings onto the surfaces of materials based on REM – Fe (Co) alloys is made difficult by their low corrosion stability, especially in electrolytic solutions, which leads to low adhesion of applied coatings.

The aim of this work is to investigate strength of the soldered joints of materials based on Nd – Fe – B and Sm – Co alloys.

Materials and research procedure

Hard magnetic alloys, chemical compositions of which have conformed to $Nd_{15}Fe_{77.5}B_{7.5}$ and $SmCo_5$ formulas, have been chosen as materials under consideration.

Smelting of all alloys has been fulfilled in a vacuum induction-arc furnace with the use of a boron nitride crucible. Homogenization of ingots has been implemented within 12 hours at a temperature of 1100 °C and residual pressure no more than 0.001 Pa.

After conditioning, ingots have been reduced in three steps. In the first stage, rough crushing of ingots into the particles up to 4 mm in size has been carried out in a jaw cruhser. In the second stage, fine mechanical grinding into the particles up to 0.8 mm in size has been implemented in a cone inertial crusher in argon medium. Thereafter the powder fraction with particles of size 50–300 μm has been separated by the screen separation method.

Final crushing been fulfilled in a vibrating ball mill in an isopropyl alcohol medium within 180–210 min until getting an average particle size equal to 3.1–3.6 μm. At this point 2% (wt.) of the Dy_2Al finishing addition powder has been added to the $Nd_{15}Fe_{77.5}B_{7.5}$ alloy powder in order to increase magnetic characteristics.

Samples have been produced by the wet extrusion method. To accomplish this, the suspension of alloyed powders in an isopropyl alcohol has been placed into a manual mold, which has been arranged between the poles of electromagnet for the extrusion time. The powder billets extrusion process has been realized in magnetic texturing field with magnetizing force more than 2500 kA/m.

Sintering of the samples after extrusion has been implemented in vacuum furnaces within 40 min at a temperature of 1120–1230 °C at residual pressure no more than 0.001 Pa. The sintered samples have been treated by mechanical operation on a flat-grinding machine to fashioning them a shape of parallelepiped with dimensions of 5×10×10 mm.

After rinse with acetone, a 5–20 μm thick copper layer has been deposited on the obtained samples by two means: galvanic and cold spraying.

For electroplating deposition there has been used the pyrophosphate electrolyte containing, g/l: $CuSO_4{\cdot}5H_2O$ – 70; $K_4P_2O_7$ – 270; KH_2PO_4 – 10. The copper precipitation mode is as follows: cathode current density is 5–6 mA/mm^2, electrolyte temperature is 35–40 °C.

Depositing of the copper coverings by cold spraying has been implemented on a Dimet-405 (Димет-405) device of powder coating with the use of C-01-01 copper powder. Compressed air with pressure of 0.6 MPa has been used as an operating gas.

Samples have been soldered over surfaces of size 10×10 mm in vacuum at a temperature of 560 °C with the use of a CTEMET 1101 amorphous ribbon solder. Thickness of solder ribbon has amounted to 0.05 mm. Surfaces of the samples have been rinsed with acetone.

Thermocycling of soldered samples has been carried out in a 12KXT-0.063-0.16 cold and heat chamber during 10 cycles. Each testing cycle has consisted of holding at temperatures of –50 and 150 °C within 1 hour at a 2 °C/min rate of achieving the given temperatures.

Corrosion testing of the soldered material samples has been fulfilled in a KTK-800 climate chamber with a holding in humid air (93%) at a temperature of 55 °C within 100 hours.

Rupture strength of the soldered joints has been measured on a P-0.5 universal tearing machine.

Quality of the soldered surfaces has been studied on a NANOME|X-160 high-resolution *X*-ray packaging inspection system with an XProAct software complex and on a Levenhuk DTX 720 digital microscope.

Results of investigations

Results of rupture strength testing of the soldered samples are presented in Table 1.

As it is seen from Table 1, rupture strength of the samples soldered over copper covering deposited by cold spraying is 1.8–2.3 times higher than that of the samples soldered over electroplating. This can be explained by the fact that in case of cold spraying, the copper powder particles accelerated in a supersonic nozzle, actively interact with substrate material (in the case in question, with rare earth alloys). On strike, particles are plastically deformed and kinetic energy is transformed into heat and partly into substrate bonding energy, providing formation of a continuous layer of densely packed particles of copper powder.

As a result, strength of the evaporated layer adhesion proves to be significantly higher than that of electroplating. If the fact that in the course of trials all the tested samples (1A–4A) has been destroyed along the boundary between the copper covering and material is also considered, then one can conclude that the soldered joint rupture strength in case of the intermediate copper coverings usage will be mainly determined by strength of the covering adhesion

Table 1

Strength of the samples after soldering

Sample number	Material	The coatication of coating method	Rupture strength σ (MPa) at a copper covering thickness of, μm		
			5	10	15
1A	Nd – Fe – B	Galvanic	26.3	24.3	19.8
2A		Cold spraying	48.4	47.8	47.2
3A	Sm – Co	Galvanic	26.7	24.5	20.1
4A		Cold spraying	48.3	48.1	47.6

since the copper and the solder rupture strength is essentially higher then the measured values.

It is interesting to note the weak correlation between adhesion strength of the covering obtained by cold spraying and the covering thickness, which on the contrary is obviously found for the coatings obtained by galvanic method. Their adhesion strength is decreasing by more than 20% as the layer thickness rises from 5 to 20 μm.

It is also needed to point out that results obtained on Nd – Fe – B and Sm – Co are very close and have similar mechanisms of changes.

As may be seen from Table 2, the soldered joints over copper covering, applied by cold spraying, are less tolerant to temperature differences. The strength drop after thermocycling at the covering thickness of 5 μm amounts to approximately 10% for samples 2B and 4B, whereas it amounts to approximately 2.5%. More obvious that difference becomes at the covering thickness of 15 μm: 12–15% and 30–42%, respectively. At the same time, break of the soldered joint of samples 1B–4B took place along the copper covering — material boundary, as for samples 1A–4A.

The adhesion durability deterioration after thermocycling is explained by great difference in linear dilatation temperature coefficients of copper ($17\cdot10^{-6}$ K^{-1}) and Nd – Fe – B ($3.4\cdot10^{-6}$ K^{-1} [13]) and Sm – Co ($6\cdot10^{-6}$ K^{-1}[13]) magnetic mediums. Therefore, the most part of temperature stresses is relaxing without adhesion breakage at low value of the covering thickness only, but mechanical strength of the covering is rising as the covering thickness grows, and this leads to the covering peeling and breakage.

As this takes place, the copper covering obtained by galvanic method, are more plastic as distinct from the coverings deposited by cold spraying. In the latter case, copper material is sstrengthened by applied shocks of particles and in fact presents the material which has been treated by cold deformation and possesses higher mechanical characteristics. As a result, the copper coverings obtained by galvanic method are proven to be steadier towards thermal loads than the ones obtained by cold spraying.

In Fig. 1 are represented *X*-ray photographs of the sample 4B before and after thermocycling with the visible defects of the copper covering, being formed after temperature influence.

At the same time, strength of the soldered joints of Sm – Co material after thermocycling is higher as compared with that of Nd – Fe – B material for all the studied values of coating thickness (Table 2), which can be explained by the higher value of a linear dilatation temperature coefficient.

Results of corrosion testing of the samples are listed in Table 3.

As is seen from Table 3, corrosion stability of the soldered joints over copper covering applied by cold spraying is significantly higher than that over covering obtained by galvanic method. The reason is that in case of galvanic method of applying coatings, there remain traces of electrolyte in pores of rare-earth metals, which become the source of corrosion.

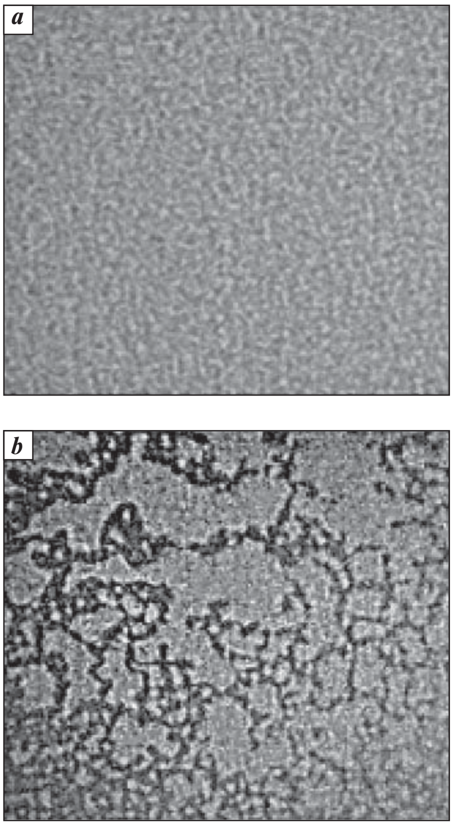

Fig. 1. *X*-ray photographs of the soldered joint 4B before (*a*) and after (*b*) thermocycling (without magnification)

Table 2

Strength of the samples after thermocycling

Sample number	Material	The coatication of coating method	Rupture strength σ (MPa) at a copper covering thickness of, μm		
			5	10	15
1B	Nd – Fe – B	Galvanic	26.1	22.3	17.1
2B		Cold spraying	44.2	37.8	27.1
3B	Sm – Co	Galvanic	26.2	22.7	17.9
4B		Cold spraying	45.9	39.1	33.2

Table 3

Strength of the samples after corrosion testing

Sample number	Material	The coatication of coating method	Rupture strength σ (MPa) at a copper covering thickness of, μm		
			5	10	15
1C	Nd – Fe – B	Galvanic	14.3	21.8	17.6
2C		Cold spraying	44.1	42.4	42.2
3C	Sm – Co	Galvanic	15.7	22.1	18.1
4C		Cold spraying	43.7	42.1	41.6

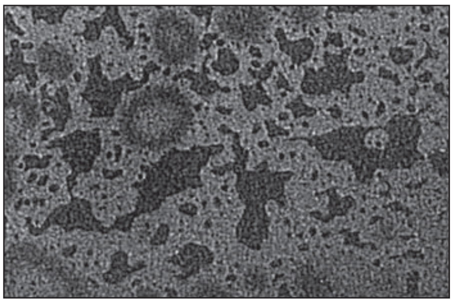

Fig. 2. *X*-ray photograph of the sample 1C after corrosion tests (without magnification)

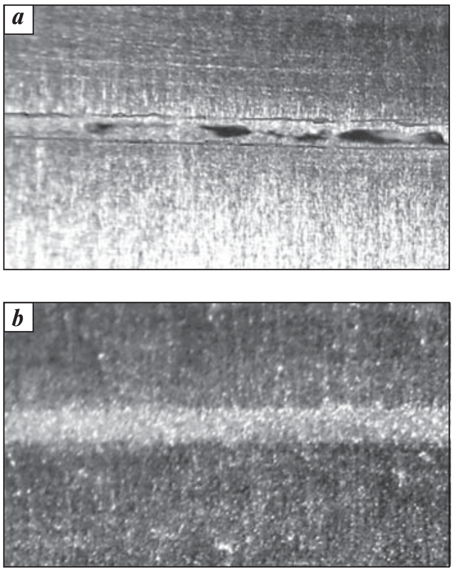

Fig. 3. Thin section of the soldered seam of the samples 1C (*a*) and 2C (*b*) with a 5 μm thick copper covering after corrosion tests (×100)

Moreover, as it has been established in the course of investigations, corrosion products penetrate to a soldered seam zone through the coating and produce the solder corrosion, if thickness of the copper covering is insufficient (5 μm and less). As a result, break of samples 1C and 3C has taken place along the soldered joint. In Fig. 2 is demonstrated an *X*-ray photograph of sample 1C with a 5 μm thick copper covering, which reflects the corrosion traces on copper covering and solder.

Adduced in Fig. 3 are the images of a thin section of the soldered seam of samples 1C and 2C, on which the crevice corrosion traces of a soldered seam are seen.

As one can see from Fig. 3, the soldered seam obtained over the copper covering deposited by cold spraying practically doesn't have the corrosion impact traces.

Hence, usage of soldering over copper covering deposited by cold spraying for materials based on Nd – Fe – B and Sm – Co alloys allows to obtain the corrosion-resistant connections with rupture strength 3–4 times higher than the glued ones. Optimum thickness of the copper covering at this amounts to 5 μm.

Conclusions

1. Rupture strength of soldered samples of materials based on Nd – Fe – B and Sm – Co alloys being soldered over copper covering obtained by cold spraying, is 1.8–2.3 times higher than that of the samples soldered over electroplating.
2. Optimum thickness of the copper covering deposited by cold spraying is 5 μm.
3. Soldering the materials based on Nd – Fe – B and Sm – Co alloys allows to obtain corrosion-resistant connections with rupture strength which is 3–4 times higher than that of the glued ones based on epoxy resins.

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Optimization of mechanical properties and hardness of cold-worked plates out of 1565ch aluminium alloy

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In light of ever increasing material requirements for most industrial applications, an ability of a supplier to guarantee a predefined level of aluminium semiproducts' properties in accordance with each customer's needs is currently taking a more prominent role in the material selection decisions. In particular, this is true for such type of a product as cold-worked (strain hardened) aluminium plates, for which certain mechanical properties and hardness distribution over the plate's thickness have to be ensured. The power of modern computing hardware and software allows to accurately resolve the tasks of processing metals by pressure that often imply high level of discretization of a geometric model into finite elements that in their turn determine the number of equations being solved. This paper presents an outcome of a research of the strain hardening process of the plates produced out of 1565ch aluminium alloy that belongs to the 5xxx series high-magnesium group of aluminium alloys. The material has been chosen for the study due to its unique combination of physical and mechanical properties as well as welding and corrosion characteristics that make it possible to use the 1565ch alloy as a structural material for a wide range of applications. The research employed both computer mathematical modeling based on finite-element analysis as well as its in-situ verification on a rolling equipment of Arconic Samara metallurgical plant. Described in the paper are the summarized results of calculation of a considerable number of separate tasks (rolling cases) in the DEFORM software that made it possible to estimate the influence of reduction schedule and other cold rolling process parameters on the final product's properties. Also presented are the results of experimental rolling trials that have proved validity and accuracy of the finite-element analysis.

Key words: finite element method, cold rolling, strain hardening, aluminium plates, 1565ch alloy.

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Introduction

Mechanical properties of pure aluminum can be improved by alloying, followed by either heat treatment (for heat-treatable alloys) or by work hardening (for non-heat-treatable alloys). The 1565ch alloy plates belong to a group of high-magnesium, strain-hardened products. In addition to high strength, the main advantage of the material is a good weldability by all types of welding [1] and high-performance under cryogenic temperatures [2]. The alloy has been successfully employed in construction of large welded structures for ship-building and railway applications [3].

The plates out of 5xxx series high-magnesium aluminum alloys are widely used for manufacturing of various high-duty parts [4]. By additional alloying it is possible to further improve mechanical characteristics of the material. The chemical composition of the 1565ch alloy used in the present study is regulated by the special technical specification [5].

In order to achieve a particularly high strength the work or strain hardening operation by cold rolling is