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Research of the protective ability of coatings on steel powder against atmospheric corrosion

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Abstract. Storing the main material for additive technologies, i. e. metal powder compositions made from steels and alloys with low corrosion resistance, is a challenge. We studied the protective ability of coatings applied by chemical heat treatment (CHT) to the surface of a steel powder, which has low resistance against atmospheric corrosion. Long-term two-year tests of powder samples with various coating compositions were carried out at natural atmospheric humidity and ambient temperature in the Northwestern Federal District of the Russian Federation. The chemical composition of the samples was evaluated before and after the test. The chemical composition and morphology of the particles were determined by electron microprobe (EMP) using a scanning electron microscope (SEM). It is shown that coatings applied by the CHT method do not create continuous protection and accelerate corrosion processes in comparison with the heat-treated powder of the initial steel.

Keywords: additive technologies, laser powder bed fusion, direct metal deposition, metal powder compositions, corrosion protection, chemical heat treatment, iodine transport method

Introduction

Additive manufacturing uses metal powders combined into common metal powder compositions (MPCs). Storing MPC steels and alloys is associated with problems due to low corrosion resistance and high chemical activity, which, during the storage of powders, can lead to corrosion damage, reducing the properties of the final products.

There is a technological problem associated with the storage of MPCs, in which partial oxidation of the surface of the powders occurs upon contact with atmospheric air of natural humidity. Corrosion damage to powder particles during production involving additive technologies is contaminated with oxides and other non-metallic inclusions, greatly deteriorating mechanical properties, especially impact strength. As a result of corrosion, the flowability of powders also decreases, meaning degradation of the main technological characteristics of the printing powder — something that is especially important for direct metal deposition (DMD) technology.

Protection of metal powders from corrosion is one of the most important practical problems in powder metallurgy and additive technologies. Due to the high dispersion and chemical activity of powdered metals, corrosion processes occur on them more intensely than on compact metals. In this case, the methods known to protect solid metals from corrosion are either not productive enough or change some properties of the metal powder.

These problems may be solved by modifying the surface of metal powders with various coatings, creating powders of the 'core-shell' type.

Surface modification can both provide protection against corrosion and/or improve other properties of the powder: for example, increase the absorption coefficient of laser radiation, which will reduce the laser power or increase the speed of 3D printing. The cladding of the surface of powder particles creates a uniform distribution of the applied elements in the resulting sample during laser synthesis, without significantly changing the chemical composition of the initial alloy.

One of the possible ways to expand the characteristics of available industrial metal powders for powder metallurgy and additive manufacturing is their surface alloying by chemical heat treatment (CHT) (Borisenok et al. 1981; Lakhtin, Arzamasov 1985; Lobanov et al. 2014; Popov 1962; Solntsev 2009; Xiaowei et al. 2005).

Previous studies have focused on the application of coatings to powder materials using iodine transport and its prospects in terms of diffusion saturation of steel and alloys (Bogdanov 2011a; 2011b; 2012a; 2012b; 2016; Khristyuk et al. 2015). Chromium coatings have been obtained on the surface of powder particles from pure iron, carbon and alloy steels (Bogdanov 2011a; 2011b; 2012a; 2012b).

The most suitable method of obtaining functional coatings on powders, especially micron and nanoscale ones, is deposition from the gas phase, including gas transport (Bogdanov 2016).

To protect the surface of low-alloy steel, it is advisable to use metals with high corrosion resistance which are suitable for application using the chemical treatment method, such as Cr, Ti or Al. To ensure high anticorrosion properties of the surface, a continuous and homogeneous coating layer should be obtained (Lobanov et al. 2014; Solntsev 2009).

Results show that chromium surface treatment improves the corrosion resistance of stainless steel due to the high concentration of chromium in the diffuse coating layer (Lee et al. 2009). Iodine (Bogdanov 2011b) or ammonium chloride has been proposed as a transport agent for a wide range of transported metals.

Corrosion tests of metal powders in various environments have shown that moisture is the decisive factor here and that corrosion processes occur exclusively at the interface between the surface of powder particles and the liquid phase. Thus, a reliable way to protect metal powders against corrosion is to block the access of moisture to the surface of the powder (Likhtman 1954).

Currently, the industry is witnessing the development of additive technologies; the use of clad powders makes it possible to obtain the necessary technological properties. Featuring prominently among them are coatings providing a high level of corrosion resistance.

The purpose of this study was to evaluate the corrosion resistance of steel powders clad with compositions based on chromium, titanium and aluminum for protection against atmospheric corrosion during storage.

Materials and methods

The basis for applying coatings using the CHT method was a steel powder of grade 45ChN2MFA (which has an average composition of 0.42–0.5% C, 0.8–1.15% Cr, 1.3–1.8% Ni, 0.2–0.3% Mo, 0.1–0.18% V), obtained using a HERMIGA 75/3 IV type melt spraying equipment. The resulting steel powder was prepared by sieving the required dispersed composition using LPBF technology (20–63 microns). A completely martensitic structure of powder particles was formed due to high cooling rates during the spraying of steel (Fig. 1a). The morphology of the particles was predominantly spherical, but there was a significant proportion of irregularly shaped particles and particles with surface defects (Fig. 1b).

The microrelief of the surface of powder particles was due to the morphology of the formed structure. A more developed rough surface was created, which may contribute to better adhesion of the coating composition.

The chemical composition from the surface of the powder particles obtained via EMP with indication of the corresponding spectra (Fig. 1) in comparison with rolling is presented in Table 1.

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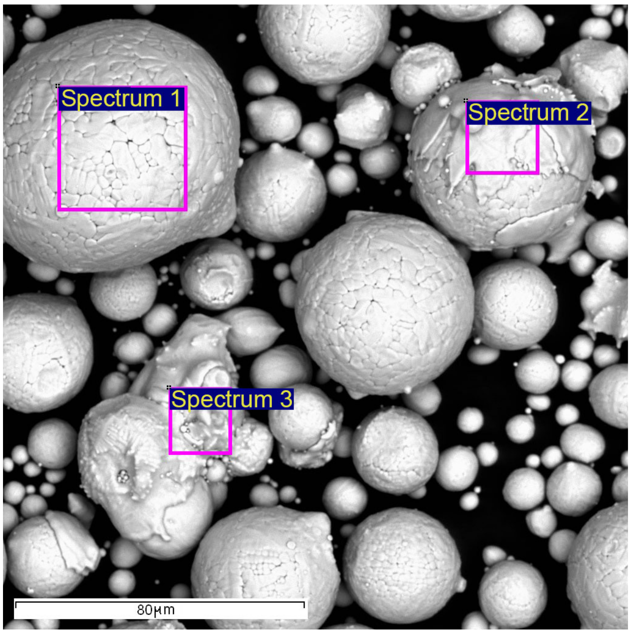

Fig. 1. Particle structure of the initial steel powder — morphology of the powder particles

Table 1. Chemical composition

Sample	Al	Si	S	V	Cr	Mn	Fe	Ni
Rolling	0.13	0.43	–	0.18	1.00	0.68	95.48	1.67
Spectrum 1	0.21	0.37	0.07	–	1.07	0.68	96.07	1.53
Spectrum 2	–	0.32	0.05	–	1.08	0.80	96.26	1.49
Spectrum 3	0.25	0.24	–	0.24	1.04	0.69	96.26	1.27

Cr, Ti and Al were chosen as cladding metals for the 45ChN2MFA steel powder based on their inherent corrosion resistance and the possibility of application by the CHT method. The compositions and parameters for CHT by the gas transport method using iodine and ammonium chloride are shown in Table 2. The optimal CHT modes were selected in order to prevent sintering of the powder. Also, to compare the corrosion resistance of the uncoated 45ChN2MFA steel powder, a powder sample was prepared after heat treatment (HT) in a vacuum furnace at a temperature of 700 °C and a holding time of 4 hours, which corresponds to the thermal cycle at CHT.

Table 2. Receiving modes

Composition of the cladding layer	Element content, mass %	Precipitation method	Temperature, °C	Time, h
Cr	5	I2	600	3
Cr	1	NH4Cl	650	3
Ti/Cr	each 0,5	NH4Cl	650	3
Ti	0,5	NH4Cl	650	6
Al	1	NH4Cl	650	6

The powder compositions were kept in a chamber protecting them from direct precipitation, but under conditions of natural atmospheric humidity and ambient temperature of the Northwestern Federal District of the Russian Federation. The samples of the powder compositions were distributed over the surface with a layer with a thickness of no more than 1 mm, which ensured the contact of the free surface of the powders with the atmosphere. The duration of exposure was two years, with the samples withdrawn for intermediate analysis after the first year of exposure. Thus, this made it possible to assess the impact of atmospheric conditions of all seasons over two years in this region.

The main criterion for assessing the anticorrosion properties of coatings was a comparison of the chemical composition of samples before and after the test. Based on the specifics of coating the surface of powder particles by the CHT-method, the chemical composition was determined by electron microprobe (EMP) using a scanning electron microscope (SEM). Due to the unevenness of the cladding coatings and the formation of locally distributed chemical compounds on the surface of the particles caused by the method of gas transport using iodine and ammonium chloride, the most representative sites were selected during the research to obtain data on the chemical composition. This method allowed us to evaluate the chemical composition of the surface of powder particles, considering its morphology. The morphology of MPC particles was studied using scanning electron microscopy methods as well as energy dispersion analysis of the content of mimic elements. Powder samples were examined before and after the exposure to a corrosive environment.

The flowability of the powders was assessed using a calibrated Hall funnel in accordance with GOST 20899-98, with bulk density determined in accordance with GOST 19440-94.

Diffuse reflection spectra of steel powders were measured. The spectra were captured using an SF-56 spectrophotometer.

The content of the mass fraction of oxygen in the powder was analyzed using a LECO TC-400 gas analyzer.

Results and discussion

Deposition of the cladding metal on the surface of the powder changes the surface roughness of the particles, affecting the flowability and bulk density of the MPC, which are important for the printing process. To assess the preservation of MPC characteristics after coating, the flowability and bulk density of the powder samples were measured (see Fig. 2, 3).

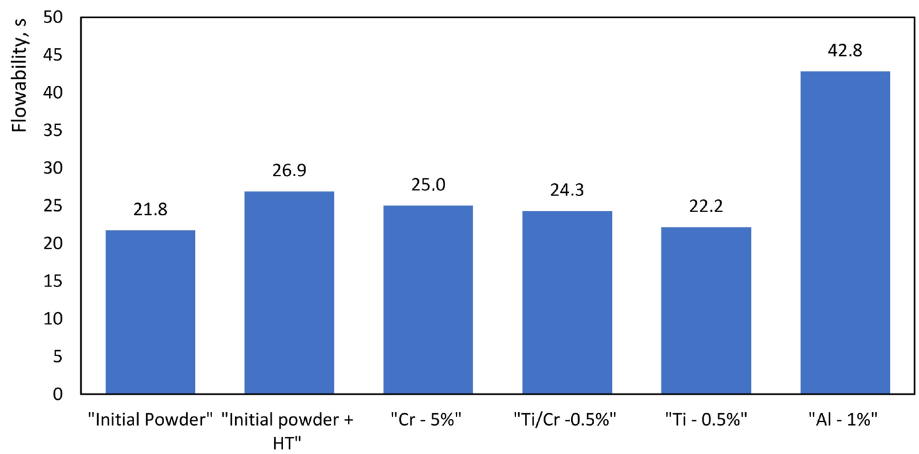

Fig. 2. Flowability of powders

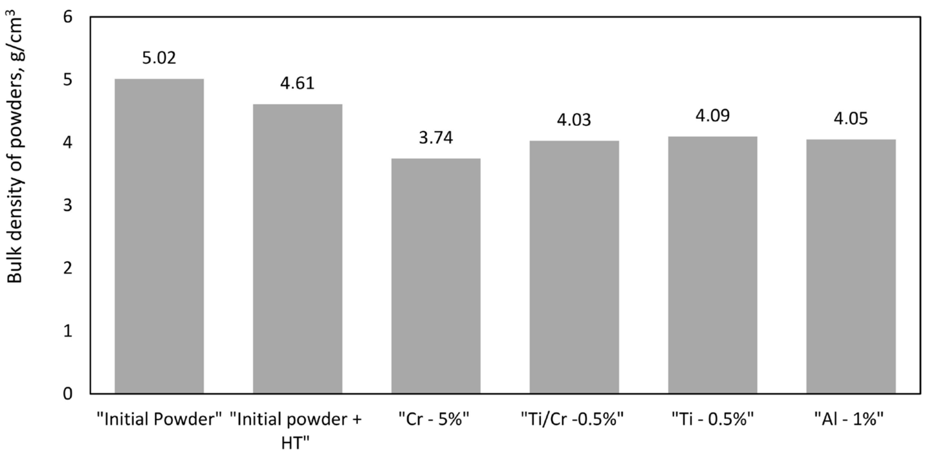

Fig. 3. Bulk density of powders

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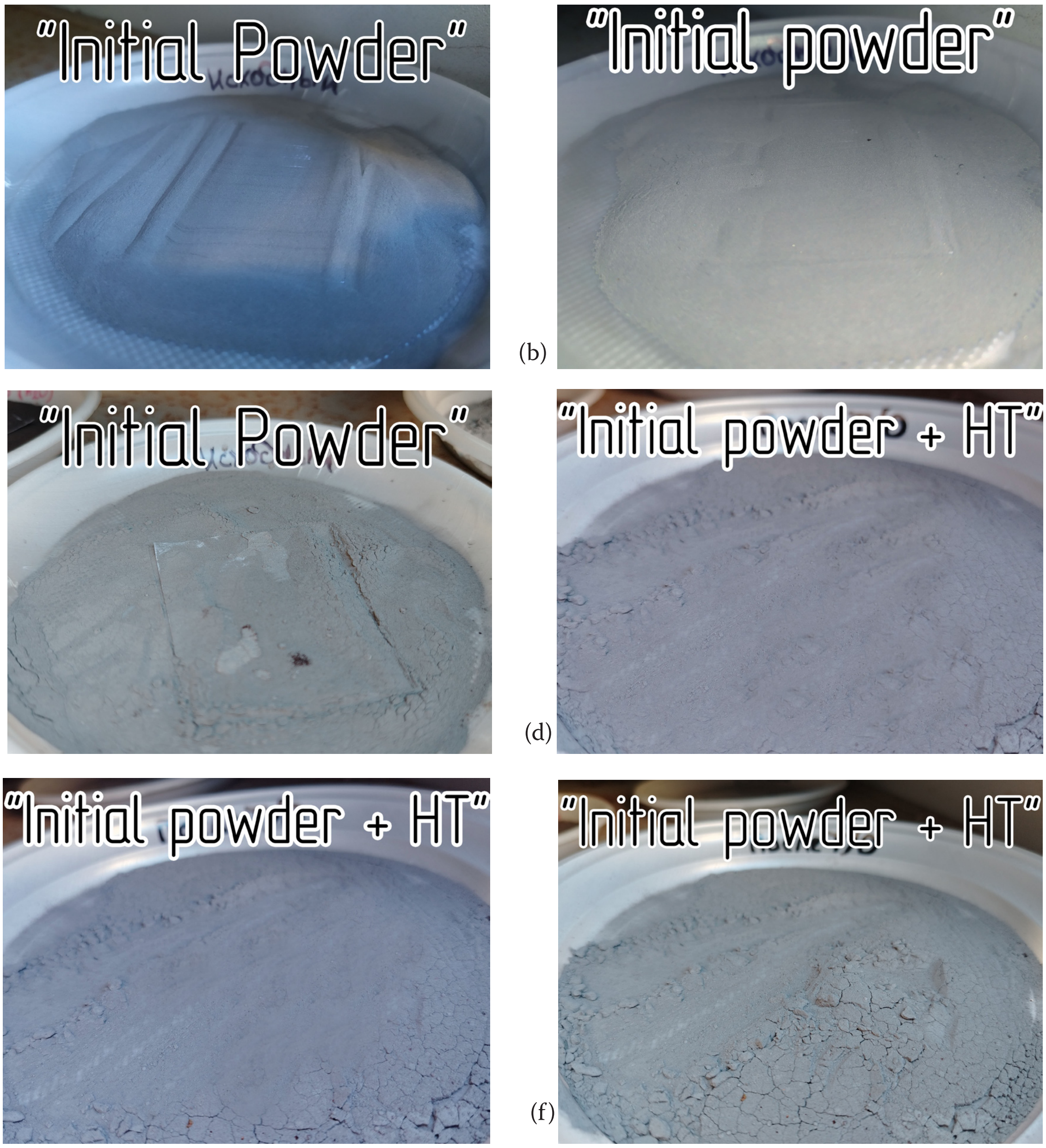
The Cr–1% sample did not have flowability according to the GOST 20899-98 method; accordingly, bulk density was not determined for this sample. The Al–1% sample was characterized by a noticeable decrease in flowability in comparison with the initial powder.

After applying cladding coatings, the flowability of the powders and bulk density decreased. MPC flowability values are especially important for direct energy and material deposition, as well as for powder bed fusion equipment with the top-feed MPC. Flowability values characterize the uniformity of application of the cladding composition and the proportion of agglomerated particles during the chemical treatment process. The values of flowability and bulk density measured for clad powders (except for the Cr–1% sample), despite the decrease in the technological qualities of the MPC, satisfy the requirements of the LPBF process and can be used to create experimental samples; however, uneven coating can critically decrease corrosion protection — to a greater extent than reduction in flowability characteristics and bulk density.

The decrease in flowability and bulk density for the powder samples under study occurred in different proportions; for example, for the Al–1% sample, a strong increase in the flow time was established, while bulk density was comparable to other powder samples. This property is determined by the processes of agglomeration of powder particles during chemical treatment, which significantly affects the flow time of the powder through a calibrated funnel, while the bulk density does not decrease significantly.

The results of a qualitative visual analysis of the powder samples with different cladding depending on test duration are presented in Fig. 4.



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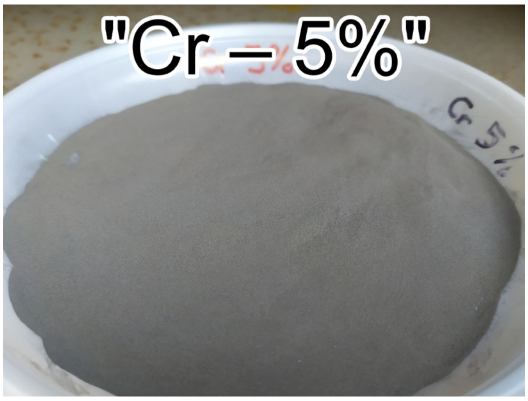

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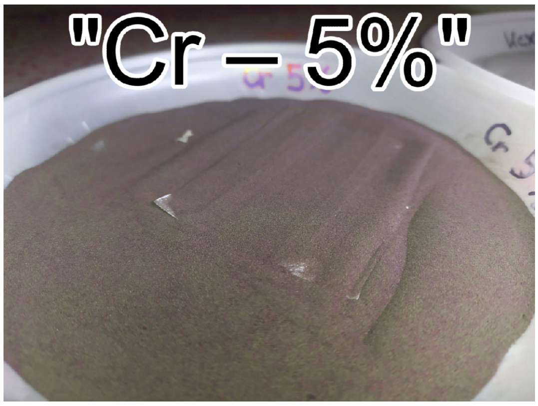

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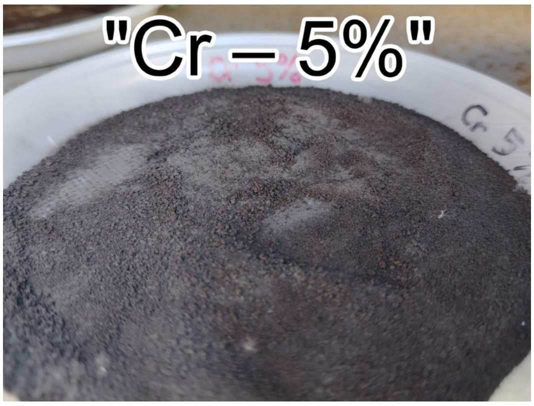

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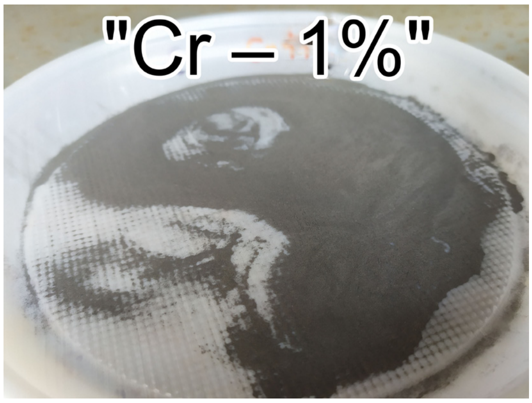

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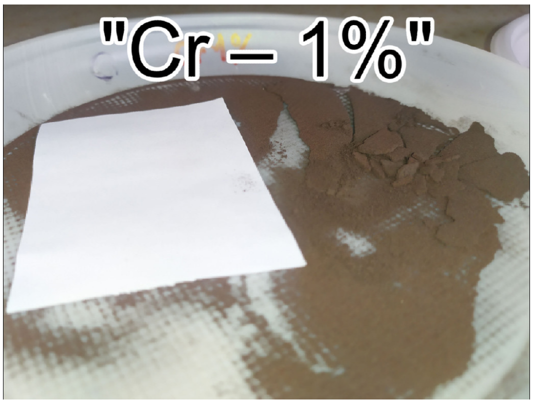

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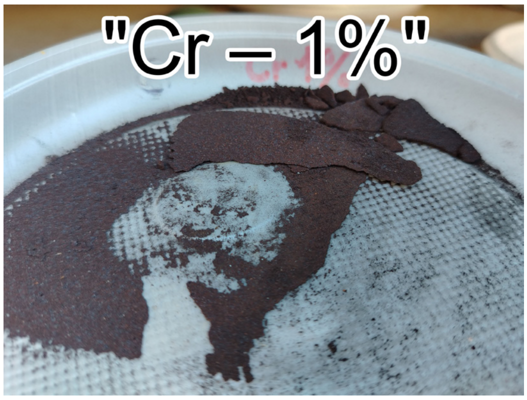

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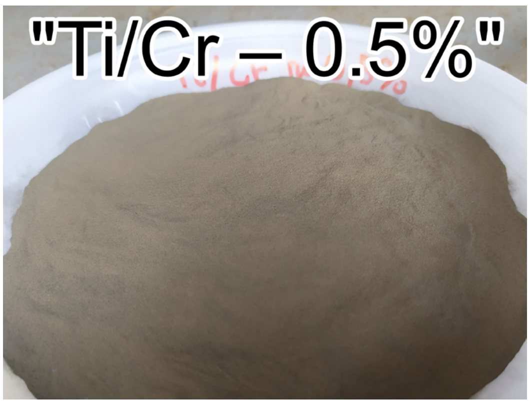

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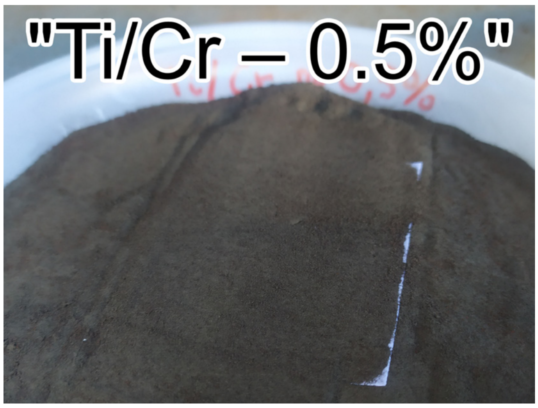

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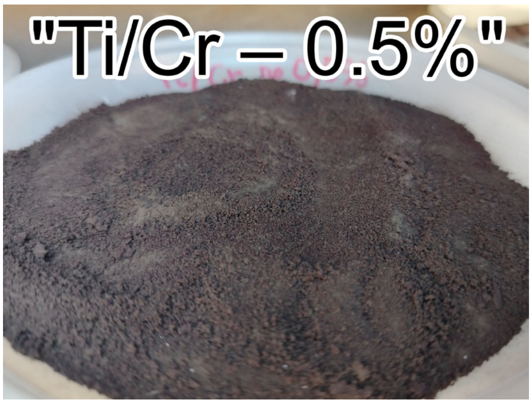

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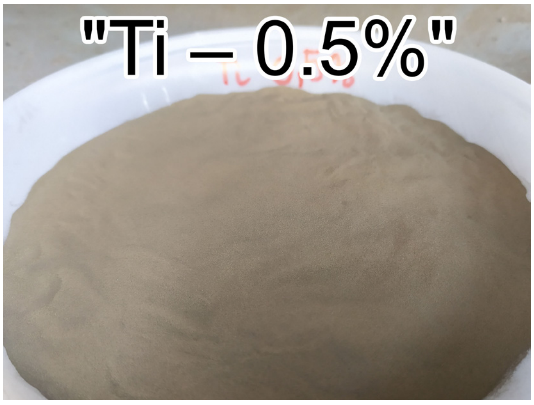

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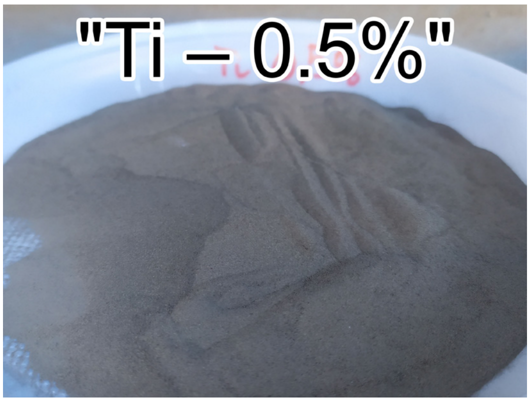

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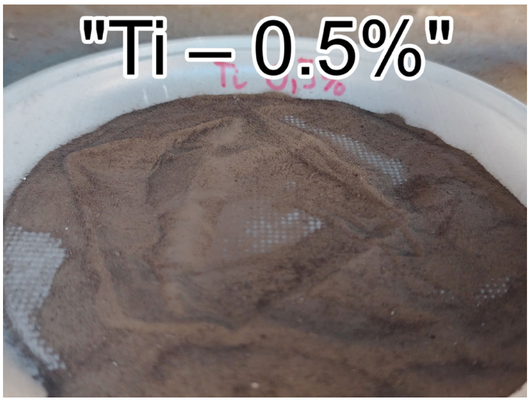

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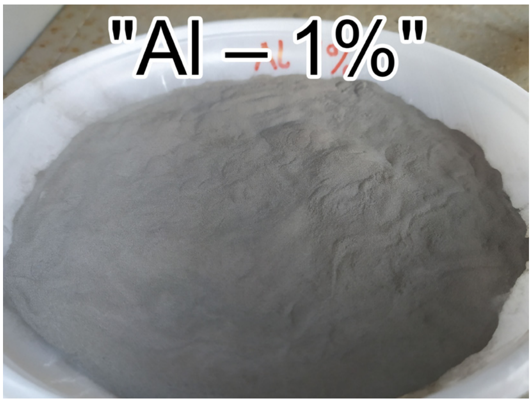

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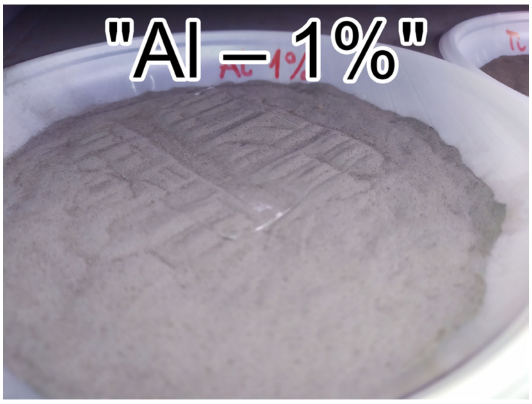

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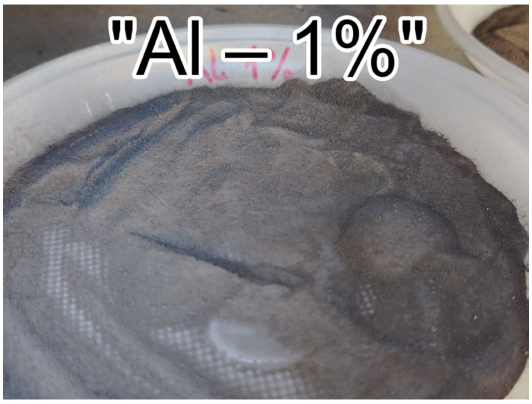


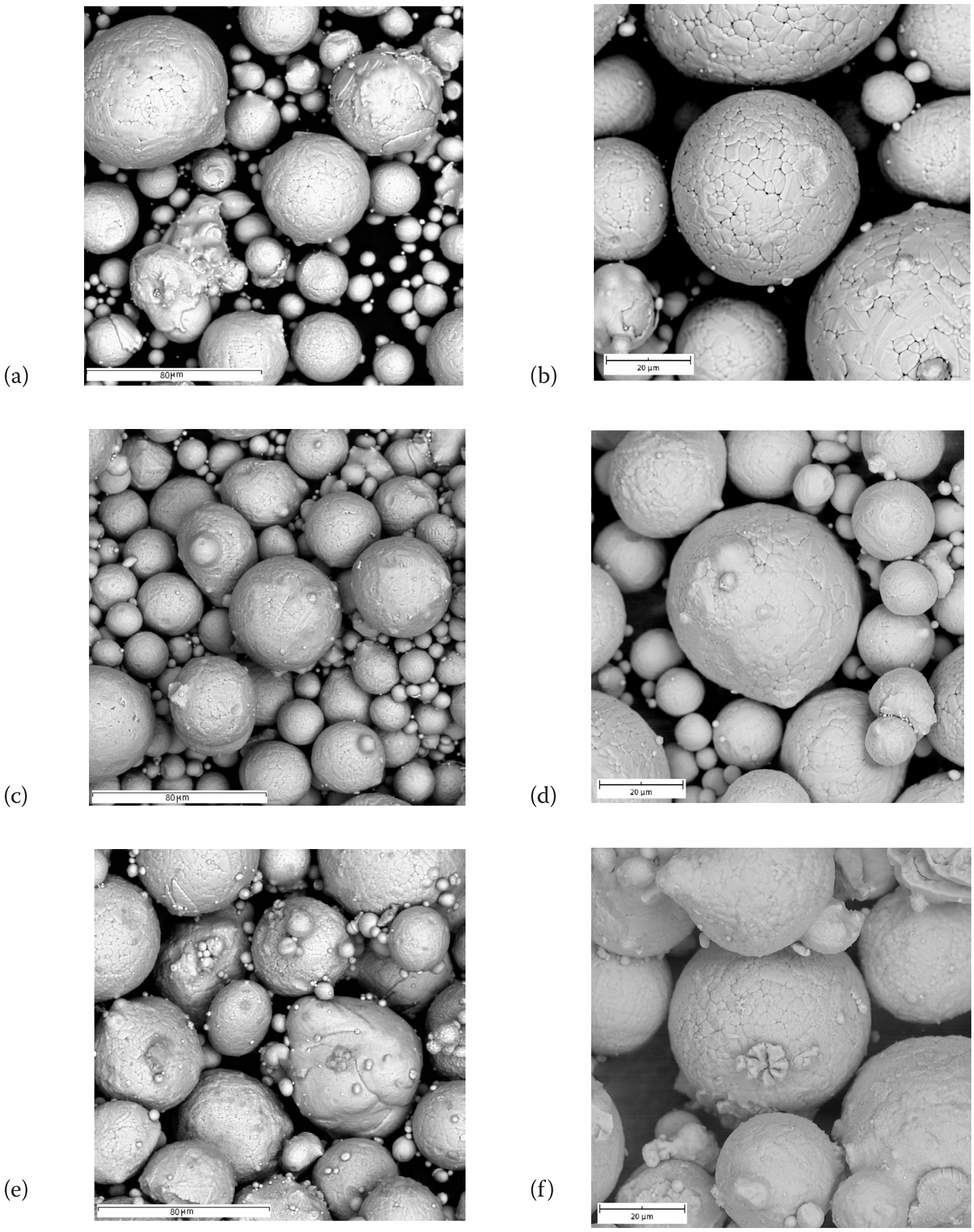
(u)

Fig. 4. Comparison of the appearance of powder samples depending on the test time (initial state, 1 year, 2 years): (a), (b), (c) — initial powder (minor agglomeration); (d), (e), (f) — initial powder + HT (minor agglomeration); (g), (h), (i) — Cr–5% (color change); (j), (k), (l) — Cr–1% (agglomeration, color change); (m), (n), (o) — Ti/Cr–0.5% (color change); (p), (q), (r) — Ti–0.5% (color change); (s), (t), (u) — Al–1% (color change)

The results of flowability and bulk density determined for the powder samples are consistent with their appearance. All the samples of clad powders are characterized by a color change; the powders acquire a red-brown color characteristic of iron oxide. The initial powder without heat treatment and with heat treatment did not change its color after testing. All the powders after the test are characterized by particle agglomeration to varying degrees.

Comparison of surface morphology and chemical composition of powder samples before and after weathering testing

For MPC samples SEM methods were used to obtain images of the surface of the powder particles (Fig. 5) before testing and after, following two years of exposure to natural atmospheric conditions.



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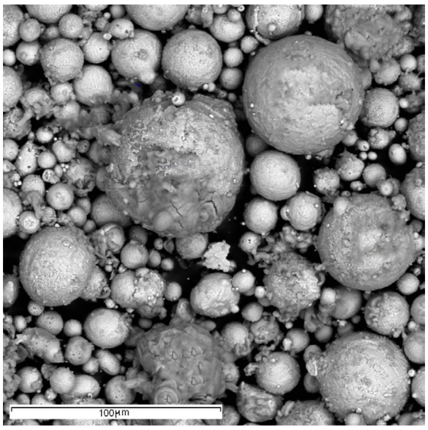

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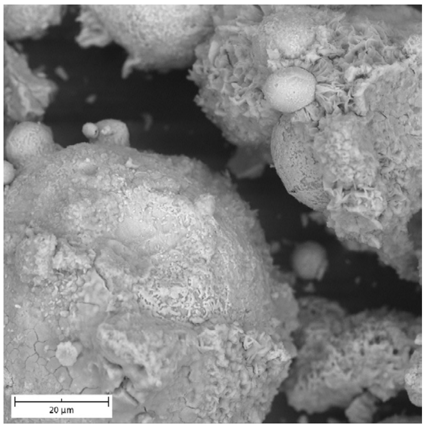

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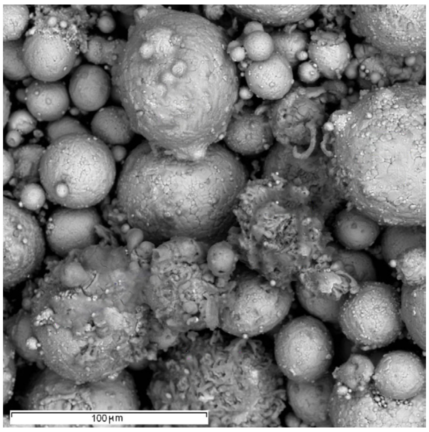

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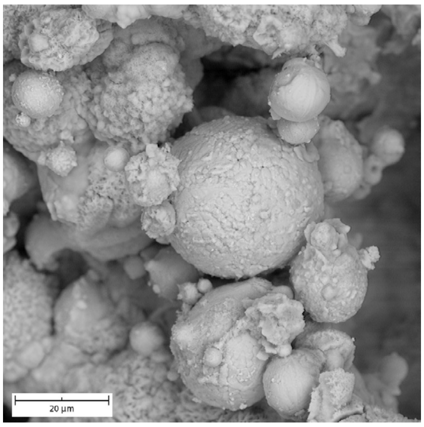

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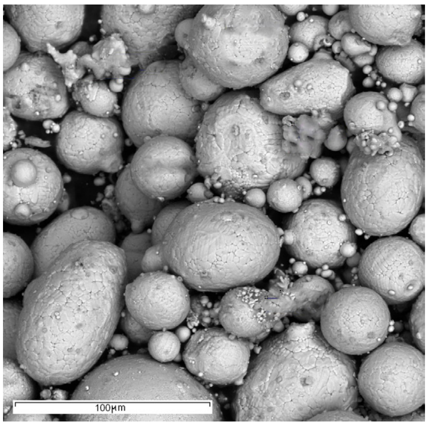

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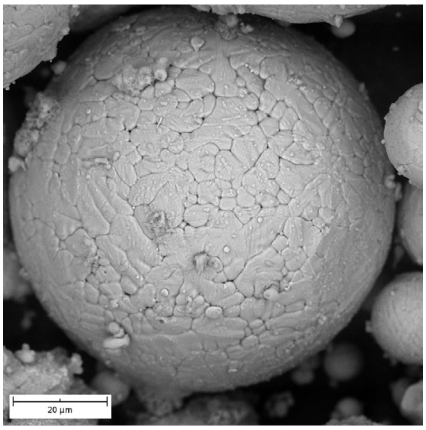

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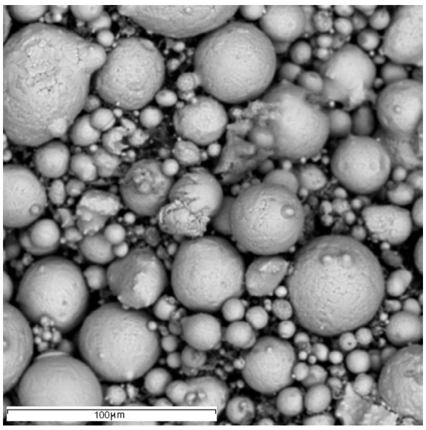

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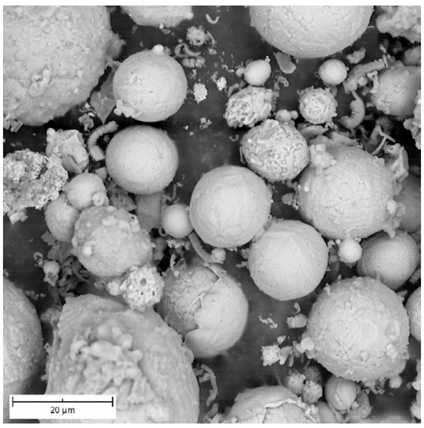

Fig. 5. Initial powder: (a) before testing, (b) after testing; initial powder + HT: (c) before testing, (d) after testing; Cr–5%: (e) before testing, (f) after testing; Cr–1%: (g) before testing, (h) after testing; Ti/Cr–0.5%: (i) before testing, (j) after testing; Ti–0.5%: (l) before testing, (m) after testing, Al–1%: (n) before testing, (o) after testing

Morphology study shows that clad powders have a layer of applied coating on their surface which is unevenly concentrated on the surface of the powder particles. Thus, no protection against corrosion is provided.

Analysis of oxygen content on the surface of the powder particles

To assess the degree of oxidation processes, the oxygen content in the powder samples was measured depending on the test time. A comparison of the oxygen concentration on the surface of the powder particles was made before testing, after 1 year and after 2 years (Fig. 6), making it possible to characterize the intensity of oxidation under test conditions.

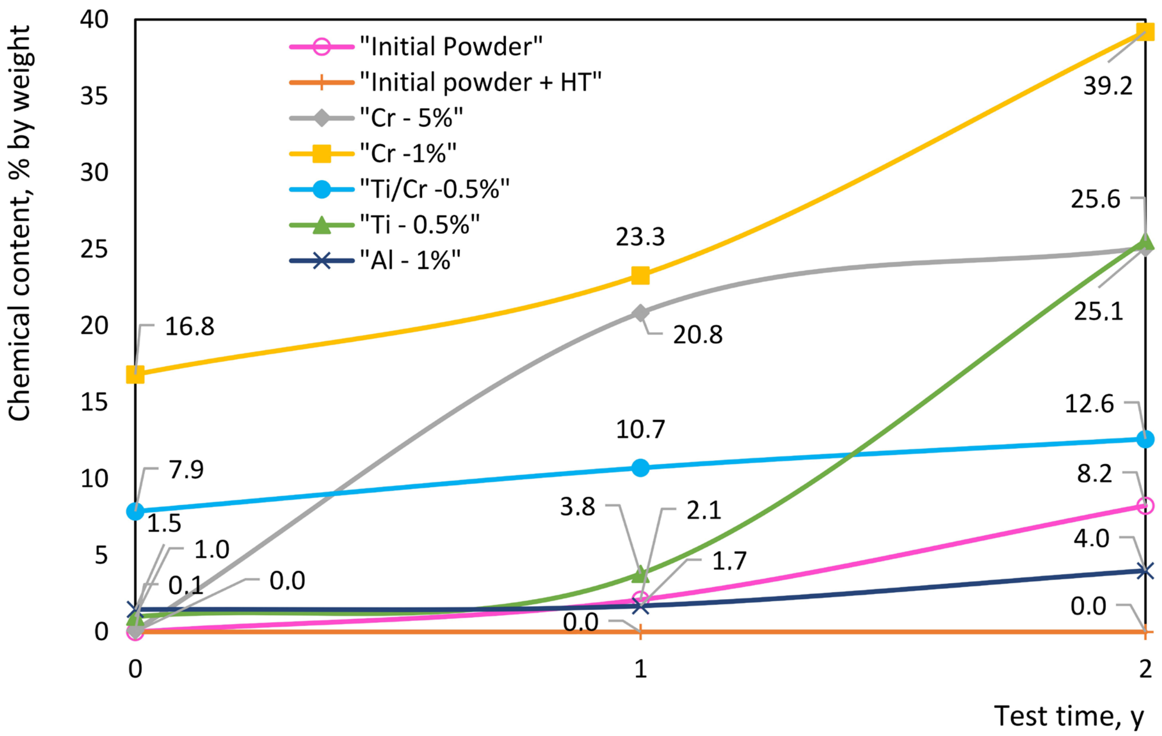

Fig. 6. Maximum oxygen content

The 45ChN2MFA steel contains a small number of alloying elements that can increase corrosion resistance. The degree of corrosion damage can be assessed by the formation of iron oxides on the surface of the powder particles.

Before testing, the powder samples were arranged in the following order according to an increase in the oxygen content: initial powder and initial powder + HT (no oxygen detected), Cr–5% (0.1% O_2), Ti–0,5% (1.0% O_2), Al–1% (1.5% O_2), Ti/Cr–0.5% (7.9 O_2) and Cr–1% (16.8% O_2). A significant mass fraction of oxygen in some of the studied powder samples before testing can characterize the quality of coating application. Apparently, when applying the compositions Ti/Cr–0.5% and Cr–1%, due to high temperatures and the presence of residual oxygen in the gas environment during chemical treatment, the powder received corrosion damage.

After testing, the samples were arranged in the following order according to an increase in the oxygen content and therefore intensity of corrosion damage: initial powder + HT (no oxygen detected), Al–1% (4.0% O_2), initial powder (8.2% O_2), Ti/Cr–0.5% (12.6% O_2), Cr–5% (25.1% O_2), Ti–0.5% (25.6% O_2) and Cr–1% (39.2% O_2). This series characterizes the ability of the powder samples under study to resist atmospheric corrosion.

Thus, the steel powder showed the best weather resistance after heat treatment; after testing, oxygen was not detected, which may apparently indicate the formation of a passivated layer on the surface of the powder particles.

For clad powders, the lowest oxygen content after testing corresponded to the Al–1% sample; the remaining powder samples were characterized by atmospheric corrosion resistance lower than that of the initial steel powder.

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It is worth noting that the samples of all the clad powders did not have a continuous surface coating of the corresponding composition; thus, the CHT method was unable to create a continuous protective layer against corrosion on the surface of the powder particles, which was confirmed by visual inspection and measurement of the oxygen concentration on the surface.

Optical properties of core-shell powders on the 45ChN2MFA steel

The absorption capacity of laser radiation with a wavelength of 1,064 nm is an important characteristic for laser synthesis technologies, as it increases the laser power utilization rate during 3D printing with these MPC powders.

After coating, the MPCs increased their absorption capacity compared to the initial powder (Fig. 7), which would allow a more efficient absorption of laser radiation power during the laser synthesis process.

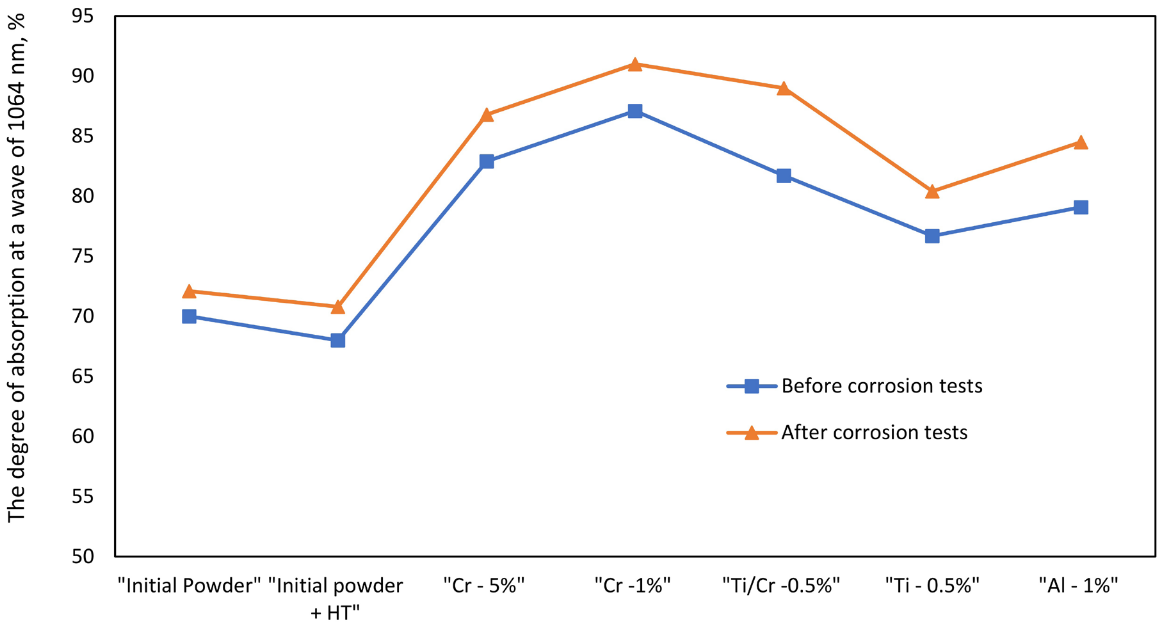

Fig. 7. Degree of absorption of clad powders before and after testing

Also, the optical properties of the clad powders indirectly characterize the formation of oxides on the surface of particles after corrosion tests. By comparing the degree of absorption of laser radiation before and after the corrosion resistance tests, we can see an increase in absorption capacity in all the powders. The absorption capacity increases with a different proportion for each composition: for clad powders this increase is more intense than for the initial and heat-treated powder.

According to test results, all clad powders have lower resistance to atmospheric corrosion than the 45ChN2MFA steel powder in the initial and heat-treated state. Of all the clad powders, the Ti/Cr–0.5%, Ti–0.5% and Al–1% samples are the most capable of resisting corrosion damage.

The high corrosion resistance of the MPC steel 45ChN2MFA in the initial and heat-treated state can be explained by the formation of a thin passivating film and slight segregation of alloying elements. The low corrosion resistance of clad powders can be explained by the uneven distribution of the cladding composition and compounds formed during the technological process of applying these coatings using the gas transport method. Thus, the surface of clad powders during chemical treatment (Khristyuk, Bogdanov 2018) experiences damage to the passivated layer obtained by spraying the initial powder due to its high chemical activity; iodine easily reacts with many metals, while their iodides are unstable compounds (Bogdanov 2012b; Rolsten 1968). Coatings do not create continuous protection and, in natural humidity, accelerate corrosion processes since the activity of metal films in the form of a coating on a substrate powder is higher than the activity of the metal powder used to obtain the coating. Films obtained by iodine transport oxidize and react with the surrounding atmosphere when the initial metal powder is still inert (Bogdanov 2012a). It is assumed that when the 45ChN2MFA steel powder is heat-treated, a thin film is created on the surface, the passivating ability of which is sufficient to provide protection against atmospheric corrosion under the test conditions.

Conclusions

1. Powders clad with various compositions have a developed porous surface with an uneven distribution of the cladding composition and compounds formed during the technological process of applying these coatings using the gas transport method. Thus, the surface of clad powders during chemical treatment experiences damage to the passivated layer obtained by spraying the initial powder or its heat treatment. The coatings do not create continuous protection and, in an atmosphere of natural humidity, accelerate corrosion processes.

2. Powders in the initial and heat-treated state during the production process — spraying the melt and subsequent heat treatment in a vacuum furnace — acquire a thin passivated layer on the surface of the particles, which is quite effective against atmospheric corrosion and does not disturb the shape of the particles.

3. After cladding, MPCs have better energy efficiency during laser melting due to a larger proportion of laser radiation absorbed by the powder.

4. Cladding of powder materials for additive technologies with various elements can be used for microalloying finished MPCs in order to change the chemical composition or uniform distribution of alloying elements prone to segregation.

Conflict of Interest

The authors declare that there is no conflict of interest, either existing or potential.

Author Contributions

All the authors discussed the final work and took part in writing the article.

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