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Heat Treatment Influence on Mechanical Properties of Nickel Based Cold Spray

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Abstract. The article deals with evaluation of Ni and NiCr Cold Spray layers as a potential method for repairing components in energy industry by replacing or refilling flawed material. Cold spray layers from two NiCr powders of different particle sizes sprayed with nozzle for inner tube application and from Ni powder of one particle size sprayed with different nozzle types – for inner and outer tube applications – were tested. The evaluation is aimed on mechanical properties and metallographic analysis of cold spray layers as received and after defined heat treatments. It summarizes tensile properties at room temperature and microstructure of used powders and final cold spray layers. Ultimate tensile strength, hardness and plastic elongation are evaluated from the mechanical point of view. Metallographic analysis is focused on ovality and size of particles in powders and of splats in final cold sprays. Porosity of sprayed layers is evaluated on tested specimens.

1 Introduction

Cold spray (CS) is a deposition process of material in form of powder, in which particles dimensions are typically of the order of micrometres. The powder is accelerated in the solid state by gas nozzle to velocities 200-1200 m/s [1]. As-sprayed CS coating has its advantageous properties, e.g. high density, hardness, increased strength and induced compressive residual stresses (potentially positive impact on fatigue and crack initiation), all with minimum thermal input to the substrate [2].

The above properties predetermine CS as a potential method for repairing components by replacing or refilling flawed material. However, it is necessary to determine the material properties of applied CS [3-5] and to prove the applicability. According to previous experience in Centrum Vyzkumu Rez (CVR), CS coatings (few millimetre thickness) exhibits sufficient strength and compactness to be tested separately without the substrate. Hence, standard tensile specimen shape is possible to use and evaluate according to appropriate standards [6].

As-sprayed CS coatings are normally characterized by high strength and low ductility [3], which lead to deterioration of fracture properties. Heat treatment (HT) can improve CS ductility for certain materials [4,5]. Hereafter, one of the determined goals is to obtain

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suitable CS coating, which can be used to repair certain pipe-components in power industry. The influence of heat treatment is thus examined in suitable temperature range considering the substrate material and for higher temperatures to evaluate the temperature influence trend.

Spraying system for industrial application should be adapted to limitations of component dimensions and work space limits. Spraying system adaptation includes non-orthogonal positioning of nozzle to substrate surface (Figure 1). Various angles of spraying can possibly have impact on CS quality. Thus the porosity and material properties are estimated for angle 45° of spraying nozzle.

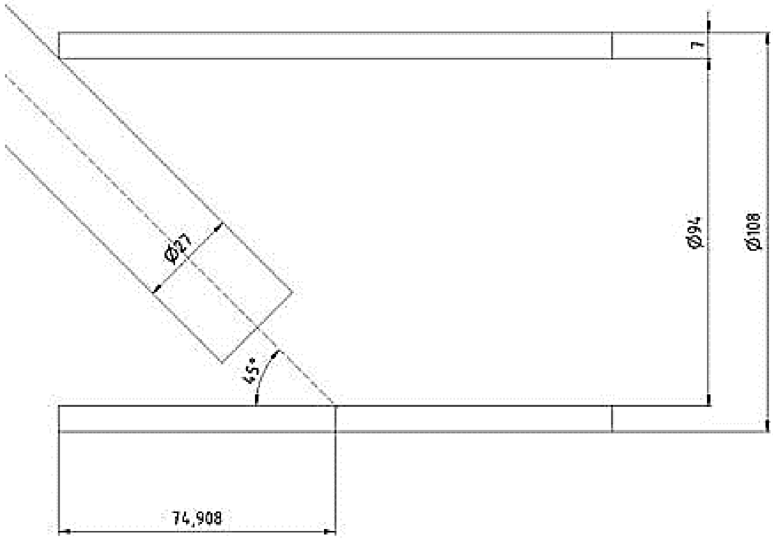

Fig. 1. Schema of CS deposition for tube 108×7 mm with nozzle angle at 45°.

The paper summarizes results of the subtask, which is aimed on verification of CS material properties. Two NiCr powders of different particle sizes sprayed with nozzle for inner tube application and Ni powder of one particle size sprayed with different nozzle types (for inner and outer tube applications) were tested.

2 Experimental procedure

As a candidate materials, nickel and NiCr (Ni 80 %, Cr 20 %) powders were used for cold spraying. Two different particle sizes (5-25 μm and 15-32 μm) of NiCr powder were used, sprayed with nozzle for inner tube application. The particle size 15-35 μm of Ni powder was sprayed with nozzles for inner and outer tube application. Non-optimal angle of the spraying nozzles was 45°, which is presumed to be used for in-situ applications. Other parameters of the spraying were guaranteed to be identical (Impact Innovations GmbH). A layer of 4.5 mm thickness was applied on aluminium substrate 65×280 mm. CS layers were cut off from substrate and used for electrical discharge machining of flat tensile specimens with 3.5×4 mm cross-section area, i.e. original 4.5 mm CS thickness was reduced to 3.5 mm.

Previous experience with NiCr CS [7] indicated low but not negligible influence of HT at 640 °C. However, potential application on tubes in energy industry allows temperature below 400 °C. Thus the HT temperature range was chosen with respect to the amount of available material from the temperature limit of considered application (390 °C) up to confirmed HT influence (600 °C). HT temperatures are summarized in Table 1. HT was performed at defined heating rate and at natural cooling rate in air. Hold time at lower temperatures was increased to support the HT influence on CS.

Table 1. Parameters of applied HT.

Designation	**Heating rate (°C/h)**	**Max. T (°C)**	**Hold time at max. T (h)**
HT1	80	390	120
HT2	80	500	48
HT3	80	600	24

Tensile tests were performed by means of ZwickRoell universal testing machine Z250. The tensile tests were conducted according to the standard [6] to determine ultimate tensile strength (UTS) and strain (A_{20}) of bone shape specimens with initial gage length 20 mm. Hardness measurement (HV1) was performed by means of Struers DuraScan hardness tester on as received CS layers and CS after heat treatment. The results are averages evaluated from at least four measurements. Metallographic analysis was performed by means of light microscopy (LM) – Carl Zeiss Observer Z1m – and scanning electron microscopy (SEM) – Tescan Mira3, focused on ovality and size of particles in powders (e.g. Figure 2) used for cold spraying and of splats (e.g. Figure 3) in final cold sprays. The analysis of powders was done from twenty measurements for each powder. The samples for analysing were prepared by floating in ethanol with aim to dissolve agglomerated structures. Porosity of sprayed layers was evaluated by means of LM on tested bone-shape specimens on the specimens' heads. The analysed area was in the middle of the CS layer due to perpendicular direction to substrate surface. Influence of HT on CS splat microstructure was not expected due to low temperatures, thus the analysis was performed only on as-received CS layers.

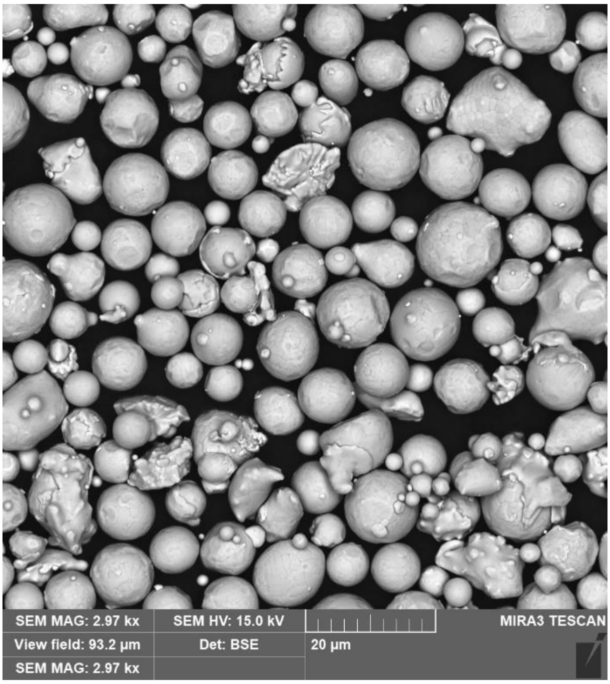

Fig. 2. SEM overview of NiCr powder with particle size distribution 5-25 μm.

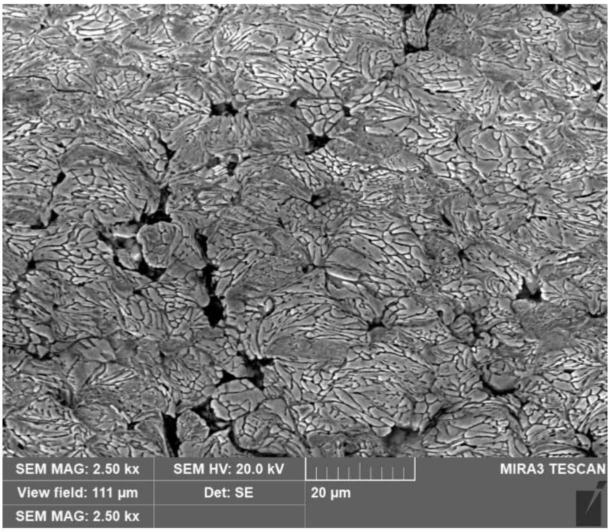

Fig. 3. SEM overview of CS layer of NiCr powder 5-25 μm sprayed by inner nozzle. CS is formed by splats with several grains. Dark areas indicate porosity of the layer.

3 Results and discussion

Particle size is generally presented by suppliers as a range of diameters (e.g. NiCr 5-25 μm), where the margins are given by sieve used for analysing. Major amount of powder, over 95 %, is in the proclaimed range. Laser diffraction analysis was not available in certificates for all powders, thus the particle size distribution was done for each powder using LM to obtain information about average particle size. Summary of particle sizes for each powder used for CS is presented in Table 2. However the proclaimed size ranges for both NiCr powders are similar, the more precise measurement reveals that the average size of one powder is significantly larger than the other one. The coarser NiCr powder is comparable with Ni powder due to the particle size. The average size of splats in CS layers is presented in Table 2. It can be noted that the size of splats should be equal to the size of powder particles independently on the used nozzle type. Comparing similar results from CS splats and powder particles gives general overview of the measurement accuracy. Table 3 contains splat size distribution in CS, which gives more precise information about sizes of used particles.

Table 2. Average particle size of powders used for cold spraying and average plat size of final CSs.

Designation	Average size ± standard dev. (μm)
NiCr powder, 5-25 μm	6 ± 3
NiCr powder, 10-32 μm	13 ± 6
Ni powder, 15-35 μm	13 ± 8
CS (NiCr 5-25 μm, inner nozzle)	10 ± 3
CS (NiCr 10-32 μm, inner nozzle)	17 ± 4
CS (Ni 15-35 μm, inner nozzle)	16 ± 7
CS (Ni 15-35 μm, outer nozzle)	17 ± 6

Table 3. Splat size distribution of CS layers.

Splat size (equivalent diameter) (%)							
μm	**0-5**	**5-10**	**10-20**	**20-30**	**30-40**	**40-50**	**50<**
CS (NiCr 5-25 μm, inner nozzle)	6	56	38	1	0	6	0
CS (NiCr 10-32 μm, inner nozzle)	0	0	81	19	0	0	0
CS (Ni 15-35 μm, inner nozzle)	7	3	61	27	1	7	0
CS (Ni 15-35 μm, outer nozzle)	2	2	69	27	0	2	0

The results from the ovality measurement of the powders and CS splats are shown in Table 4. The presented results of ovality representation are an arithmetical average obtained from three measurements from each powder and CS layer. It is obvious that the initial high ovality of the powders is deformed to lower ovality of CS splats, as expected. The ovality of splats in NiCr CS is similar for both layers made from powders of different particle sizes. Ni powder used with two different nozzles results in lower ovality of splats sprayed with outer nozzle, which is in agreement with the expectation of higher quality CS than CS sprayed with inner nozzle. Higher deformation of splats in Ni CS compared to NiCr CS is due to identical parameters of spraying caused by properties of material itself.

Table 4. Results of percentage ovality representation of powders particles and CS splats.

Designation	**Ovality representation (%)**				
	0-0.2	**0.2-0.4**	**0.4-0.6**	**0.6-0.8**	**0.8-1**
NiCr powder, 5-25 μm	1	5	14	15	66
CS (NiCr 5-25 μm, inner nozzle)	0	10	42	38	10
NiCr powder, 10-32 μm	0	3	8	13	75
CS (NiCr 10-32 μm, inner nozzle)	0	11	44	37	7
Ni powder, 15-35 μm	0	3	11	20	66
CS (Ni 15-35 μm, inner nozzle)	1	34	40	24	0
CS (Ni 15-35 μm, outer nozzle)	4	45	42	7	2

Mechanical properties together with porosity are summarized in Tables 5 - 8 for each CS layer as received and after HT (see Table 2). Note that elongation A_{20} was not evaluated for CS from NiCr powders (Tables 5 and 6). The elongation was negligible due to brittleness of the NiCr layers and it can be considered as zero. The porosity and pore sizes are evaluated for as received material and heat treated material too. There was no expectation of influence of HT on porosity, however, the influence of porosity have to be excluded in case of scattering results for specimen series after various treatments. The low scatter of porosity results of each CS layer and similar pore sizes are observed comparing one material with and without treatment, therefore each CS layer can be considered as a homogeneous material.

Table 5. Results of mechanical properties and porosity of CS (NiCr 5-25 μm, inner nozzle).

Designation	**UTS (MPa)**	**A_{20} (%)**	**HV1**	**Porosity area (%)**	**Pore size (μm^2)**
as received	124±16	N/A	334±21	1.6	676
HT1 (390°C, 120h)	157±23	N/A	400±23	2.2	434
HT2 (500°C, 24h)	182±32	N/A	386±25	1.9	344
HT3 (600°C, 12h)	198±29	N/A	279±12	1.8	354

Table 6. Results of mechanical properties and porosity of CS (NiCr 10-32 μm, inner nozzle).

Designation	**UTS (MPa)**	**A_{20} (%)**	**HV1**	**Porosity area (%)**	**Pore size (μm^2)**
as received	67±11	N/A	254±29	7.7	2064
HT1 (390°C, 120h)	92±21	N/A	304±30	7.6	2523
HT2 (500°C, 24h)	118±19	N/A	308±33	8.7	2538
HT3 (600°C, 12h)	125±11	N/A	228±16	7.4	2017

Table 7. Results of mechanical properties and porosity of CS (Ni 15-35 μm, inner nozzle).

Designation	**UTS (MPa)**	**A_{20} (%)**	**HV1**	**Porosity area (%)**	**Pore size (μm^2)**
as received	75±7	0	204±11	1.4	1146
HT1 (390°C, 120h)	204±7	1.9±0.7	105±8	0.9	917
HT2 (500°C, 24h)	199±4	2.5±0.5	101±6	1.1	769
HT3 (600°C, 12h)	213±5	5.3±1.7	95±3	0.8	955

Table 8. Results of mechanical properties and porosity of CS (Ni 15-35 μm, outer nozzle).

Designation	**UTS (MPa)**	**A_{20} (%)**	**HV1**	**Porosity area (%)**	**Pore size (μm^2)**
as received	508±45	0	220±10	0.2	410
HT1 (390°C, 120h)	378±8	17.0±2.0	106±7	0.1	269
HT2 (500°C, 24h)	384±6	21.6±2.6	114±12	0.2	486
HT3 (600°C, 12h)	396±2	28.6±3.2	104±4	0.1	389

Considering previous result [7] of NiCr layer sprayed by outer nozzle perpendicular to surface (i.e. UTS approx. 400-500 MPa without HT), the values of UTS were expected to be higher for NiCr layers. However, the high absolute value of porosity together with low ductility result in fracture in early stage of tensile loading. The influence of porosity can be seen by comparing the UTS results of NiCr CS (Tables 5 and 6), where the CS from the coarser powder shows a higher porosity and larger pores, which leads to lower UTS values. The influence of the nozzle type on porosity can be observed in Tables 7 and 8, where the outer nozzle results in significantly lower porosity. Generally, the UTS values scatter for as received material and the scatter of results decreases with ductility increase, which is observed on Ni CS (Tables 7 and 8). The scatter of UTS results persists for NiCr layers, the influence of HT on ductility was negligible (Tables 5 and 6).

Negligible ductility of as received CS layers is generally expected due to the high residual stress caused by deformation of powder particles during the spraying process. Brittleness removement due to HT depends on the material of used powder. NiCr layer is obviously more thermal stable, however, the influence of HT is still significant and it increases with higher temperatures. Small increase of ductility is expected, though it have to be confirmed with additional measurement with higher accuracy of deformation measurement and it is only hypothetical at the moment. On the other side, Ni layer is significantly loosing residual stress even for low temperature of HT, which leads to significant ductility increase at the expense of UTS (see Figure 4) and HV1. For quantifying residual stresses, additional analysis is recommended. Hardness decrease is significant especially between the heat treated and as received material. Dependance on HT temperatures cannot be affirmed due to scatter of similar results for Ni layer. Comparing influence of HT in Ni CS (Tables 7 and 8), it can be noted that the ductility improvement due to HT results in UTS increase in cases, where UTS is significantly low due to porosity (Table 7).

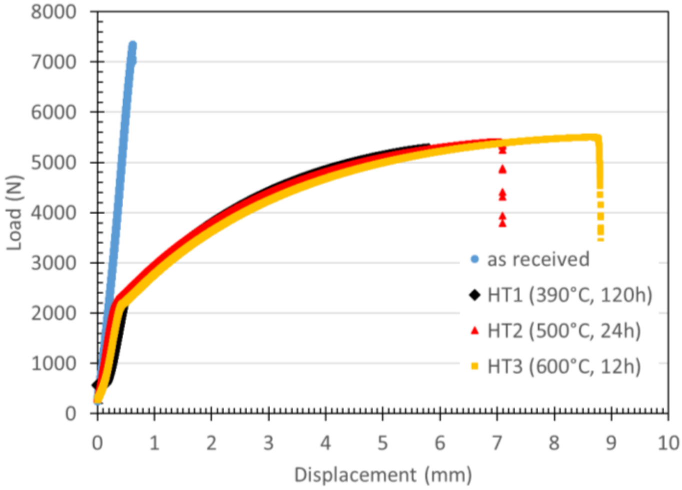

Fig. 4. Load-displacement tensile curves of Ni layers (outer nozzle). HT results in UTS decrease and in elongation increase with increasing HT temperature.

4 Conclusion

Tensile and hardness tests were performed on four different CS layers differing in used powder (Ni and NiCr) and nozzle type, supplemented by metallographic analysis of powders particles, CS porosity and CS splats. Three heat treatments in temperature range 390-600 °C were performed on each layer, on selected series of specimens.

The type of the nozzle used for cold spraying influences the final porosity of the layer. Optimizing spraying parameters for the nozzle for inner tube applications is recommended, however, that is not the aim of the study. Spraying powder with lower diameter of particles improves cold spray properties and results in lower porosity.

The porosity has significant influence on ultimate tensile strength values. The cold spray as received is generally brittle. The brittleness in combination with porosity leads to noteworthy decrease of ultimate strength.

The influence of heat treatment on tensile properties was observed on each cold spray. It differs with used material of powder. NiCr behaves as thermally stable material, heat treatment influence results in increase of ultimate strength, though the influence on ductility have to be confirmed by additional measurement. Heat treatment of Ni layers results in significant decrease of ultimate strength, however, it has positive impact on ductility. The ductility grows with increasing temperature of heat treatment. The ductility improvement due to heat treatment results in ultimate strength increase in cases, where ultimate strength is significantly low due to porosity.

The ovality of splats in cold spray layer appears to be material dependent parameter. The results of splat ovality representation for NiCr is equal for both powder particle sizes. NiCr is generally harder material and shows lower rate of deformation than Ni splats. Using outer and inner nozzle with Ni powder resulted in different values of splat ovality. Comparing the absolute sizes of powder particles and equivalent diameters of splats results in similar values.

Negligible ductility of as received CS layers is generally expected due to the high residual stress caused by deformation of powder particles during the spraying process. Removing the brittleness due to heat treatment depends on the material of used powder and it is expected to be connected to the loss of the residual stress. Measurement of residual stresses is recommended for further analysing of heat treated cold sprays.

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