Wear resistance of HVOF sprayed coatings from mechanically activated thermally synthesized Cr$_3$C$_2$–Ni spray powder

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Abstract. In the current study a Cr$_3$C$_2$–Ni spray powder was produced by mechanically activated thermal synthesis. The following aspects were studied: (a) production and characteristics of spray powders, (b) spraying and characteristics of coatings by HVOF, and (c) abrasive wear resistance. A HVOF spray system Diamond Jet Hybrid 2700 (propane hybrid gun from Sulzer Metco) was used for deposition. Coating thickness was approximately 350–400 $\mu$m. The structure and composition of the coatings were determined by SEM and the phase composition by XRD methods. Coating surface hardness and microhardness in the cross-section were measured. Abrasive rubber-wheel wear (ARWW) and abrasive erosive wear (AEW) were tested. The wear resistance of the coatings produced from an experimental powder was comparable to that of a similar commercial one.

Key words: chromium carbide, spray powder, mechanically activated thermal synthesis, HVOF spray, wear resistance.

1. INTRODUCTION

Application of the thermal spray technology has been increasing rapidly [1]. Thermal spray processes, especially the high velocity oxy fuel (HVOF) spray, provide excellent wear resistant coatings for different industries like aviation, pulp/paper, oil/gas, and metal processing [2–5]. To ensure competitive advantages and to increase the market share, supporting equipment of thermal spray (manipulating systems, robots, computer controlled systems), better combinations of particle velocity and temperature as well as feedstock powders, which contribute significantly to the running costs, are being developed [6,7]. To increase spraying efficiency and produce dense high quality coatings, desired feedstock materials should be spherical and equally distributed in shape and size [8].

Over the last decades Cr$_3$C$_2$ coatings produced by the HVOF spray have become increasingly more popular and exceed WC–Co coatings in industrial areas where heat, oxidation, and corrosion resistance are required. On the other hand, the cost of these feedstock materials is relatively high due to their complex composition. Therefore, the price of powder production may be a factor for selecting other powders or technologies.

Reactive sintering (also called mechanically activated synthesis (MAS) or integrated mechanical and thermal activation (IMTA)) is the process that has been developed and used successfully for producing bulk hardmetal/cermet materials from WC–Co, Cr$_3$C$_2$–Ni, and TiC–NiMo with promising results [9–11]. In that process the initial powders are first activated mechanically, for example in a ball mill, and then thermally synthesized by sintering. Carbides are formed during the thermal process, although some formation of carbide can be noticed already in the mechanical activation phase. The purpose of mechanical...
activation in powder metallurgy is to use lower sintering temperatures [7].

The aim of this study was to produce Cr$_3$C$_2$–Ni spray powders for wear resistant applications via mechanically activated thermal synthesis (MATS) and compare the properties of experimental coatings with similar commercial ones.

2. EXPERIMENTAL MATERIALS AND METHODS

2.1. Powder production and characterization

Experimental spray powders were produced from 99.5% pure chromium (Pacific Particulate Material) with an average grain size 7 μm, carbon black KS6, and nickel powders with a particle size 2–3 μm. A mixture consisting of 11.45 wt% C, 20 wt% Ni, and 68.55 wt% Cr was prepared. The selected carbon content and temperature were to help avoid free carbon in the structure after sintering. Powders used for spraying were manufactured according to the procedure shown in Fig. 1. Mechanical activation was conducted in a ball mill in an ethanol environment. The milling time was 72 h, and the ball-to-material ratio was 15 : 1. The mill and the balls were made of WC–Co hardmetal. Thermal synthesis was carried out in a conventional vacuum sintering environment at 1100 °C and holding time of 30 min. It was followed by mechanical milling to obtain feedstock powder with a particle size of 20–63 μm for HVOF deposition.

Powder granularity was determined by a particle size analyser Analysette 3 PRO and the particle shape was determined with a scanning electron microscopy (SEM) apparatus Zeiss EVO MA-15. For X-ray analysis (EDS) an Oxford Instruments INCA Energy system was used. The phase of the synthesized powder was identified using the X-ray diffraction (XRD) method with Cu Kα radiation (Bruker AXS D5005).

2.2. Spraying of coatings

Carbon steel C45 with dimensions 100 mm × 25 mm × 5 mm was used as the base material for coatings. Coatings were deposited by a HVOF spray system Diamond Jet Hybrid 2700 (propane hybrid gun from Sulzer Metco) from Tampere University of Technology. The commercial powder Cr$_3$C$_2$–25NiCr (Amperit 588.074), widely used in industrial wear resistance applications, was selected as a reference. The parameters of the HVOF spray are shown in Table 1. Prior to spraying, the steel substrates were grit blasted by using alumina with mesh 36 to improve coating adhesion. Coatings were deposited layer by layer (about 50 μm per pass) to obtain the final coating thickness of 400 μm.

2.3. Microstructure and hardness of coatings

Polished coating cross-sections were subjected to optical microstructural examination by a light microscope (OM) using an Omnimet image analysis system and SEM Zeiss EVO MA-15. The Oxford Instruments INCA Energy system was used for EDS to estimate the composition of coatings.

Surface Vickers hardness (HV) measurements were performed at a load of 9.8 N (1 kgf). Microhardness in the cross-section was measured using a Matsuzawa MMT-X device at a load of 2.94 N (300 gf). This load was selected to obtain the size of indents comparable with the sizes of the hard phase in the composite. On both occasions, Vickers indentor was used and the standard deviation (STD) of the measurements was calculated.

2.4. Abrasive wear testing

The coatings were tested for abrasion based on the abrasive rubber-wheel wear (ARWW) test. The diameter of the ring was 228.6 mm, the applied force 130 N, the feed rate of the abrasives 330 g/min, and the speed of rotation 200 1/min (linear velocity 2.4 m/s). The testing time was 5 min.

The abrasive erosive wear (AEW) of the coatings was tested at the impact angles 30° and 90°. The velocity

Table 1. Spraying parameters

| Parameter                  | Value |
|----------------------------|-------|
| Propane flow, L/min        | 68    |
| Oxygen flow, L/min         | 240   |
| Air flow, L/min            | 383   |
| Carrier gas flow, L/min    | 12.5  |
| Powder feed rate, g/min    | 60    |
| Spray distance, mm         | 230   |
of abrasive particles was 80 m/s and the feed rate of abrasives was 600 g/min. The testing time was 10 min.

Quartz sand with a particle size of 0.1–0.3 mm was used as the abrasive. The relative volumetric wear resistance to steel C45 (normalized, 200 HV30) was calculated at ARWW and AEW based on the volume wear rates of the reference steel C45 and the studied coatings.

3. RESULTS AND DISCUSSION

3.1. Characterization of spray powder

Powder particles produced by MATS and then mechanically milled were irregular in shape and size (Fig. 2). In addition to particles of 20–63 μm, the powder contained very small particles coming from mechanical milling. The same effect was observed in an earlier study with WC–Co powders [12].

On the XRD patterns two main phases Ni and Cr$_3$C$_2$ were identified (Fig. 3). Some of the spectrum peaks are somewhat broadened, giving evidence of residual stresses and crystal lattice defects. Quantitative composition of the powder was also calculated from the XRD patterns. The results are presented in Table 2.

3.2. Characterization of HVOF coatings

The thickness of the sprayed coatings determined by the SEM analyses of the cross-section of the investigated images was in the range 350–400 μm (Fig. 4). Micro-

![Fig. 2. Shape and size of powder particles produced by MATS.](image)

![Fig. 3. XRD patterns of Cr$_3$C$_2$–20Ni powder produced by MATS.](image)

![Table 2. Composition of Cr$_3$C$_2$ based spray powders](table)

| Type of powder | Chemical composition, % | Particle size, μm |
|----------------|-------------------------|------------------|
| Experimental Cr$_3$C$_2$–20Ni | 75.7 22.7 1.5 | +20–63 |
| Reference Cr$_3$C$_2$–25NiCr (Amperit 588.074) | 75 25 | +15–45 |

![Fig. 4. Coating structures: (a) Cr$_3$C$_2$–Ni; (b) Amperit 588.074.](image)
hardness of the sprayed coatings was the same for experimental and reference coatings. However, the surface hardness of the experimental coating was around 1.5 GPa lower than that of the commercial coating (Table 3) due to a softer matrix (Ni vs NiCr, respectively).

### 3.3. Abrasive wear resistance of coatings

#### 3.3.1. Wear resistance

Table 4 shows the results of abrasive wear tests. At the ARWW test, the experimental coating showed 7.7 times higher wear resistance than the reference steel C45 and its resistance was almost the same as that of the coating produced from the commercial powder (Table 4).

At the low impact angle AEW test (α = 30°), the wear resistance of the experimental coating was 1.5 times as high as that of steel C45 and approximately the same as that of the commercial reference coating Amperit 588.074 (Fig. 5). However, in the high impact angle AEW test (α = 90°), both the experimental and the commercial coating showed poor results compared to the reference steel C45. This agrees with the results of earlier studies of AEW [13].

#### 3.3.2. Wear mechanism

Due to grinding in the wearing-in stage of the ARWW test, smoothing of the surface takes place. As it follows from the topographical image (Fig. 6), some wear traces can be seen in the area of pores and/or inclusions.

Topographical images of the eroded surfaces of the experimental coating at a low impact angle (α = 30°) and at a normal impact angle (α = 90°) are shown in Fig. 7a and 7b, respectively. As can be seen, differences in the wear mechanism at the studied impact angles are insignificant. At the low impact angle, the traces of microcutting (Fig. 7a) and at the normal impact angle, some ploughing of the surface and traces of direct removal of hard particles can be seen (Fig. 7b).

### 4. CONCLUSIONS

- The results of the study of powder production demonstrated that the mechanically activated thermal synthesis (MATS) technology can be used to produce feedstock materials for the HVOF spray.
- The HVOF sprayed coatings obtained from the experimental powder are competitive with the coatings from analogous commercial powders: the microhardness of the experimental coating was the same as that of a similar commercial powder.
- In the wear tests, the results of the experimental coatings were relatively similar to those of the coatings produced from the commercial powder.

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**Table 3. Thickness and hardness of HVOF sprayed coatings**

| Type of coating     | Thickness, μm | Vickers hardness HV, GPa |
|---------------------|---------------|--------------------------|
|                     | Surface       | Cross-section            |
|                     | HV1           | HV0.3                    |
| Experimental Cr3C2–20Ni | 400           | 7.3 ± 0.80               | 10.1 ± 1.48 |
| Reference Cr3C2–25NiCr | 350           | 9.6 ± 1.08               | 10.1 ± 1.22 |

**Table 4. Wear rates at ARWW and AEW tests of HVOF coatings**

| Type of coating     | Wear rate, mm³/kg |
|---------------------|-------------------|
|                     | ARWW              | AEW (α=30°) | AEW (α=90°) |
| Experimental Cr3C2–20Ni | 2.1           | 24.9/93.1     |
| Reference Cr3C2–25NiCr | 2.0           | 22.1/64.3     |
| Reference C45        | 29.4             | 35.7/27.2     |

**Fig. 5. Relative wear resistance of sprayed coatings to steel C45.**

**Fig. 6. Topography of the wear surface after ARWW of Cr3C2–Ni.**
In the wear studies the coatings from the experimental powder showed better results at abrasion than steel C45. At abrasive erosion, the wear resistance of the experimental and commercial coatings was slightly higher at a low impact angle wear as compared to steel C45; at a normal impact angle, the HVOF sprayed coatings studied did not work: their relative wear resistance was about 0.3–0.4.

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Fig. 7. Topography of eroded experimental Cr3C2–20Ni coatings: (a) α = 30°; (b) α = 90°.
Mehhanoaktiveeritud termosünteesitud kulumiskindlad pinded Cr$_3$C$_2$–Ni
pihustuspulbritest

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Kroomkarbiidi baasil kiirleekpihustuspindes on volframkarbiidsete kõrval leidmas ühe laiemat kasutust eelkõige
valdkondades, kus kuumus- ja korrosioonkindlus on olulised. Kuid tulenevalt keerulisest koostisest on nimetatud
pulbrid kallid.

Käesoleva uuringu eesmärgiks on alternatiivmeetoditega saadud pinnete saamine. Need on omadustelt võrreldavad
tööstuslikult toodetavatest pulbritest pinnete.

Lähtekomponentideks Cr$_3$C$_2$–Ni kermispulbri saamisel olid Cr, C ja Ni, saamismooduseks mehaaniliselt akti-
veeritud termosüntees (mechanically activated thermal synthesis, MATS). Töö vaadeldi järgmisi küsimusi: a) pulbri
saamine ja iseloomustamine, b) pinnete pihustamine ja omaduste uuring, c) pinnete abrasiivkulumise uurimine.

Pihustamiseks kasutati kiirleekpihustussüsteemi Diamond Jet Hybrid 2700 (Sulzer Metco propaan-hübriidseade). Pin-
nete paksus oli vahemikus 350–400 μm. Pinnete struktuuri ja koostist uuriti SEM- ning XRD-meetoditega ja
määramat pinde pinnas kõvad ning mikrokõvad ristlõikes. Pindeid uuriti abrasiiv- ja erosioonkulumise tingimustel.
Saadud eksperimentaalpinnete kulumiskindlus oli võrreldav tööstuslikest pulbritest pihustatud pinnete.

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