ČESKÉ VYSOKÉ
UČENÍ TECHNICKÉ
V PRAZE

FAKULTA
STROJNÍ

TEZE
DISERTAČNÍ
PRÁCE

ČESKÉ VYSOKÉ UČENÍ TECHNICKÉ V PRAZE

FAKULTA STROJNÍ

ÚSTAV STROJÍRENSKÉ TECHNOLOGIE

TEZE DISERTAČNÍ PRÁCE

RISK FACTORS OF TRIBOLOGICAL PROCESSES AND THEIR ENVIRONMENTAL IMPACT

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Doktorský studijní program: Strojní inženýrství

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Oponenti:

Teze byly rozeslány dne: ....

Obhajoba disertace se koná dne .... v ……… hod.

v zasedací místnosti č. 17 (v přízemí) Fakulty strojní ČVUT v Praze, Technická 4, Praha 6

před komisí pro obhajobu disertační práce ve studijním oboru Strojírenská technologie.

S disertací je možno se seznámit na oddělení vědy a výzkumu Fakulty strojní ČVUT v Praze, Technická 4, Praha 6.

Prof. Ing. Jan Suchánek, CSC.

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Fakulta strojní ČVUT v Praze

1. SOUČASNÝ STAV PROBLEMATIKY

As our modern industry undergoes a push towards optimization and increased efficiency, a necessary growing becomes more desirable feature within the industry to follow up market needs.

To guide these improvements and being sure that it will fulfill standards and regulations, there will be important factors need to be controlled, and one of these factors is tribology and its side effects.

However, many advanced methods have been proposed in the field of tribology, concentrating on two main factor: friction and wear resistance. Modern technologies can be in some cases quite over-engineered or too complex especially when scientific committee proposing new methods, such as lubricating oil new composites or surface treatment by using new materials.

Over the last few decades there has been considerable activity in the field of risk assessment. This has mainly taken place in international organizations such as European Chemicals agency.

A high motivation has been noticed here and will be considered in this dissertation, by presenting and concentrating on new invented materials, which can be used in tribological applications as robust future material in industrial usage, and showing clearly advantages and disadvantages of this usage.

As this dissertation is focused on cermets as promising material to be used in tribological applications, the aim is to summarize and estimate risk factors, produced by combining Nickel alloys 625 and Boron Carbide particles in plasma powder transferred arc welding (PPTAW).

Till today's date, Cermets, including Ni625 and B_4C, are nowadays reaching a great level of development, to prove its optimal properties, Boron Carbides (B4C) ceramic, as high temperature resistance and hardness material and Inconel® metal, as corrosion resistant material, high yield and tensile, high temperature strength, excellent weld ability and creep strength.

Each of the various tribological processes, by which material can be lost from a surface in service, leaves its fingerprint both in the topography of the worn surface and in the size, shape and number of the particles, which make up the wear debris. The size of material removal from work piece surface as well as the rate of material removal are the prime dependent outcome parameters, which depend on the wear-mechanism taking place during tribological operation.

Cermets optimal properties bring improvement to tribological properties, wear resistance and friction, as advantage. Nevertheless, from other hand, general overview for disadvantages, which could be brought by this usage, need to be considered here by risk assessment preparation according to European Chemical Agency standards and classifications.

The necessity to study this risk came from lack of knowledge and experience with this new combination of cermets (Ni625 and B4C), that is why collecting particles, produced by tribology test, for confocal microscope analyses, is necessary for this research, to distinguish micro/ nano particles existence. Electron microscope analyze was performed to confirm cobalt/ Nickel particles existence in free micro/ nano particles, which is considered as hazard, allergic particles for human being and prohibited material to be found in micro/ nano size particles [1]. According to European chemical agency Nickel can be announced clearly as dangerous material.

Cobalt substance is registered under the REACH Regulation and is manufactured in and / or imported to the European Economic Area, at ≥ 10 000 tonnes per annum.

This substance is used by consumers, in articles, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

Talking about Cobalt and it's existence in industry, this substance is used in the following products: metals and metal surface treatment products. Other release to the environment of this substance is likely to occur from: indoor use and outdoor use resulting in inclusion into or onto a materials (e.g. binding agent in paints and coatings or adhesives

According to European chemical agency, Cobalt considered as Dangerous material. "According to the harmonised classification and labelling (ATP14) approved by the European Union, this substance may cause cancer, may damage fertility, is suspected of causing genetic defects, may cause long lasting harmful effects to aquatic life, may cause an allergic skin reaction and may cause allergy or asthma symptoms or breathing difficulties if inhaled."

Additionally, the classification provided by companies to ECHA in REACH registrations identifies that this substance is fatal if inhaled, is very toxic to aquatic life with long lasting effects, may damage fertility or the unborn child, is harmful if swallowed, causes serious eye irritation, may cause damage to organs through prolonged or repeated exposure and is suspected of damaging fertility or the unborn child.

Therefore, risk assessment and diagnostic aid in assessing the health of operating plant, which may contain many tribological contacts, requires not only appreciation of the mechanisms, by which wear occurs, but also careful and standardized procedures for debris extraction and observation either by solving these particles in liquids or using cleaning oils to prevent having hazard materials in free movement atmosphere.

2. CÍLE DISERTAČNÍ PRÁCE

Nowadays, the environment pollution produced by metal traces is a highly concerned issue. One of the important causes of trace metal pollution is particles discharging out of industrial processes which normally exist in micro/ nano size particles.

These metal traces pose life-threatening effects on human kind health and safety. The aim of this dissertation is to determine cobalt (Co) and Nickel (Ni) existence which are produced from cermets, consist of boron carbide and Ni 625 alloys, used in tribology applications to evaluate Co and Ni toxicities in these samples.

According to previous studies and occupational exposure to cobalt mostly occurs via inhalation as part of the refining, alloy production and hard metal industries, but dermal exposure is also possible. It is known that different forms of cobalt can have different toxicological effects [25].

Cobalt is a technically important metal, used mainly as a binder in hard-metal industry and as a constituent of many alloys. Boron Carbide and Nickel alloys compounds are used as case study in this dissertation.

Implantation and preparation of cobalt alloys has been prepared by plasma powder transferred arc welding, and cobalt compounds induced local and sometimes metastasizing sarcomas in life being. An indication of possible harmful effects of cobalt alloys or compounds in human populations has arisen from medical use, in hard-metal industries, and at cobalt production.

Unfortunately, having boron carbide and nickel alloys has a major problem, and the size of most of the investigated populations has been rather small, so none of the investigations alone gives sufficient evidence of a carcinogenic effect in humans, but taken together there is an indication of a potential risk that should be explored further [26].

Nickel is one of the most common allergens causing allergic contact dermatitis worldwide. The aim of the study is to evaluate the contributing factors to nickel contact allergy (NiCA) and it's great negative impact on working atmosphere in tribological areas.

Main target out of this dissertation is to proof possible risk of using these cermets in tribological applications by finding, during tribological experiments, that cobalt and Nickel particles produced and this can be dangerous for health of people working in this field.

Secondary objective is to further investigate the tribological behaviour of cermets B4C/ Ni alloys 625, as a promising composite to reduce friction, material loss and increase wear resistance.

Expected findings from this study:

1. Existence of cobalt and Nickel micro & nano particles resulted from tribology.

2. Heat treatment effect on cermets physical characteristics and how it will change tribology efficiency.

3. Micro & nano sized cobalt and Nickel particles existence in free movement.

4. Conclude the effectiveness of the approach as a means of showing risk of this material usage and possible impurities during manufacturing processes.

3. METODY ZPRACOVÁNÍ

- Inconel 625 Samples Preparation: samples were deposited by plasma transferred arc (PTA) using the Nickel-based super alloy Inconel 625, manually controlled procedure, with a commercially available plasma hard-facing automate PPC 250 R6 (KSK, s.r.o., Czech Republic) in a tight chamber. Used machine is designated for glass industry applications, mainly for hard-facing of glass mold edges. The hard-facing automate can work with plasma current pulses up to 200 Hz between 50 and 250A with 30 percent B4C and 70 percent Ni625 alloys [16] [A.7].

- Temperature Measurement During Samples Preparation: Manufacturing of 3D-parts by plasma transferred arc (PTA) powder deposition process involves repeated heating and cooling processes at the same location causing thermal distortion and residual stresses in the substrate and deposited material. Samples' temperature were tracked during this preparation, so that a clear understanding clarified by temperature diagram, showing which tempo-behaviour can be shown here in a diagram and how it will affect samples quality. Therefore, pre-heating was used to the working piece and measured by Tempo measuring points T1, T2, and T3. These measuring points where attached to samples according to figure 1. Welding procedure took from 2 to 6 min. During this waiting time the temperature of the work piece increased rapidly to reach maximum limit approx. 900oC on T1 point [15].

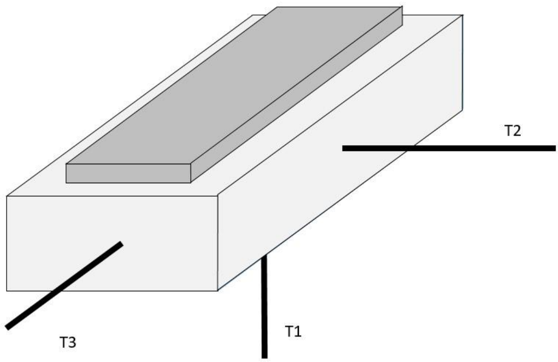

Figure 1: Attached Temperature Measuring Points on testing samples

- Testing Samples Heat Treatment Before Tribology Test: Heat Treatment is necessary for the precipitation of strengthening phases and improvement of the mechanical properties.

Step 1 aging	Heat to 720ºC during 180 min, hold for 8h, furnace cool to 620 ºC, hold for 8h, air cool
Step 2 solution	Heat to 980ºC during 180 min, hold for 1h, air cool

- Polishing and Samples Preparation For Tribology Test: Polishing/grinding for samples' surface was done to make surface more smooth and suitable for testing applications.

- Tribometer Tribology Test:

Tribometer or Tribotester is a generic name for a device which is used to simulate friction and wear at the interface between surfaces in a relative motion under controlled conditions.
In this testing phase it will be needed to have tablet on plate (Disk) Linear Reciprocating. For this test we need to have Disk and Tablet. Tablet will be fixed with applied load on it, while disk connected to motor to give the Linear Reciprocating movement. See Fig.5.22.
One Disk was cut by using cutting machine square shape 1 [cm^2]. See figures 5.23.

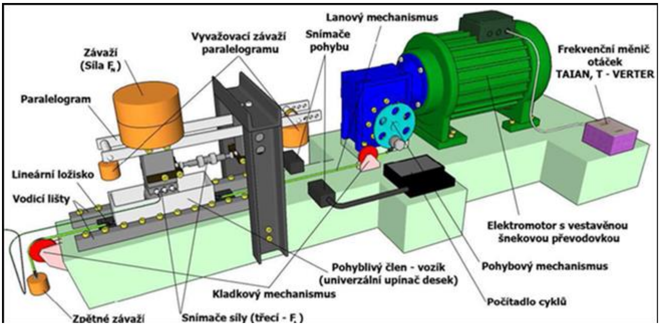

Figure 5.22: Tribometer tribology machine

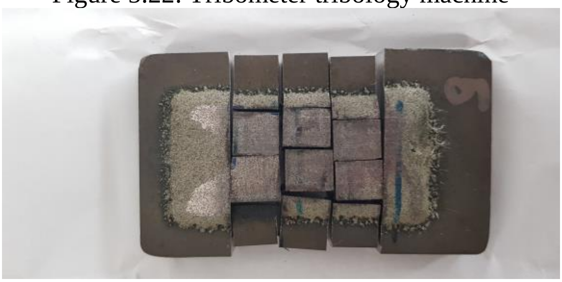

Figure 5.23: Cutting disk with cutting machine square shape (1 cm^2)

- Tribometer test performed into these parameters:

1. Applied load on disk: 3 [Kg].

2. Speed: Stroke (Step): 2*35 [mm]

Main target of this test is to measure tablet weight during test running to recognize how much material we lose and how big is wear rate for this material. First study case, heat treated tablet vs heat treated disk. Test lasted for 1335 [min] and during this time 15 measurements were conducted for tablet's weight. Second study case, non heat treated tablet vs heat treated disk. Test lasted for 1620 [min] and during this time 13 measurements were conducted for tablet's weight.

Heat treated tablet vs Heat treated disk: worn-out material as average 0.00036/ 120 [min].

Non Heat treated tablet vs Heat treated disk: worn-out material as average 0.00026/ 120 [min], [A.7].

- Confocal micoscope:

This study demonstrates the potential of confocal laser scanning microscopy (CLSM) as a characterization tool for different types of micro particles. Micro-particles were prepared by collecting particles produced from tribometer testing equipment.

This technique can be used to determine the shape and outer structure of the microparticles; nevertheless, It has certain limitations when used for the analysis of the internal structure of such particles [28].

Observed photos and analyze from this technique will give remarkable traces for micro & nano sized particles existence.

The following figures 5.33 and 5.34 projection view produced from a series of optical sections provides important information about a three-dimensional specimen than a multidimensional view, including mapping and distance for dimensional points.

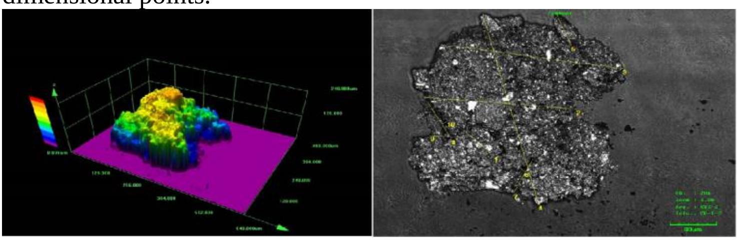

Figure 5.33: Confocal analyze for particle1

Dimension point	Judge	Length[um]	dZ[um]
1		75.6801	4.336
2		272.7893	-26.032
3		326.21	-68.088
4		399.6017	94.836
5		68.6843	41.58
6		10.6213	-46.676
7		77.4198	-117.988
8		84.823	49.74
9		40.3877	-12.572
10		9.3942	46.012

Figure 5.34: Confocal analyze for particle1 measurements

- Electron microscope and X-ray particles mapping:

SEM is the most widely used instrument in routine materials characterization for its advantages of exibility in sample preparation, easy data interpretation, and multiple functions of imaging, chemical analysing, and the recently developed electron back scattered diffraction (EBSD). For dozens of years, SEM has been one of the essential tools in failure investigation, including comprehensive analyses of wear debris, worn samples, and subsurface micro-structure.

When electrons enter the specimen, the electrons are scattered within the specimen and gradually lose their energy, then they are absorbed in the specimen. This behaviour is shown in Fig.5.47. The scattering range of the electron energy, the atomic number of the elements making up the specimen and the density of the constituent atoms. As the energy is higher, the scattering range is larger, the scattering range is smaller 3. Electron microscope measurement steps involves accelerating voltage 15KV, which is considered as the e_ective voltage in this case study, surpassed by a charged particle along a defined straight line. Two types of photos have been taken by electron microscope.

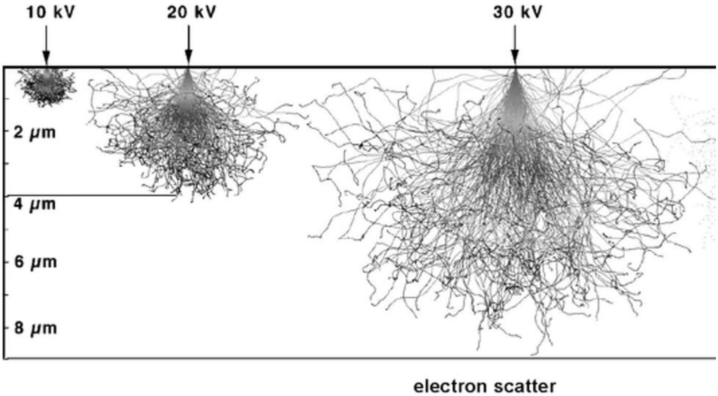

Figure 5.47: Simulation showing the scattering behaviour of electrons within the specimen.

X-ray mapping is used to obtain the distributions of speci_c elements. In this analysis, the electron probe is scanned over a speci_c area and characteristics X-ray with specific energies are acquired.

From figure 5.48, show X-ray mapping for particles from tribometer test under electron microscope. Particles are from tribometer test combination non-heat treated tablet vs heat treated disk.

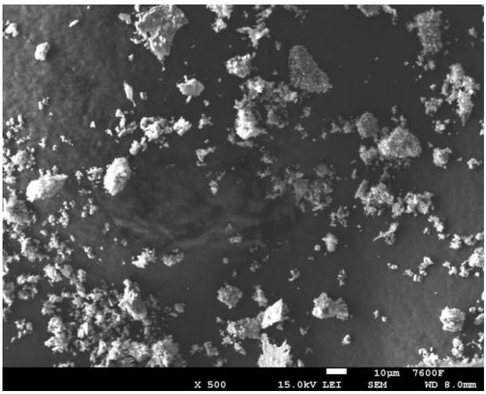

Figure 5.48: LEI 10µm scale, non-heat treated tablet vs heat treated disk; Analysis of particles by means of three SEM modes, the SEM SE imaging mode showing nano sized particles.

- Chemical Mapping by backscattered electrons:

Backscattered electrons are those scattered backwards and emitted out of the specimen. When the incident electrons are scattered in the specimen. They are sometimes called reected electrons. Since backscattered electrons possess higher energy than secondary electrons, information from a relatively deep region is contained in the backscattered electrons. The backscattered electrons are sensitive to the composition of the specimen.
The atomic number of the constituent atoms in the specimen is larger, the backscattered electrons yield is larger. That is, an area that consists of a heavy atom appears bright in the backscattered electron image. Thus, this image is suitable for observing a compositional difference.

Scanning was done by electron microscope equipped with EDS (Energy dispersive X-ray spectroscopy), detector Oxford X-Max 50[mm^2] for chemical analysis (element mapping).
If the specimen surface has irregularity, the intensity of backscattered electrons becomes higher in the direction of specular reection. The feature can be used to observe the topography of the surface.
Therefore, chemical analyze by using electron beam will show chemical map for materials existence in particles shape, which can move freely in testing atmosphere and surroundings.

For the variant non heat treated tablet & heat treated disk, these figures 5.60, 5.61 show visible particles by electron microscope are the following: Chromium , Nickel, Iron, Molybdenum, Cobalt, Copper, Zinc, Silica, Boron, Carbon, and Oxygen. [A.7]

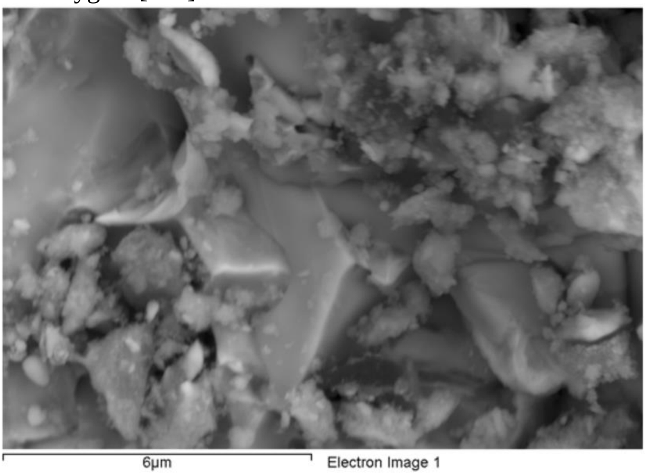

Figure 5.60: Visible particles by Electroscope

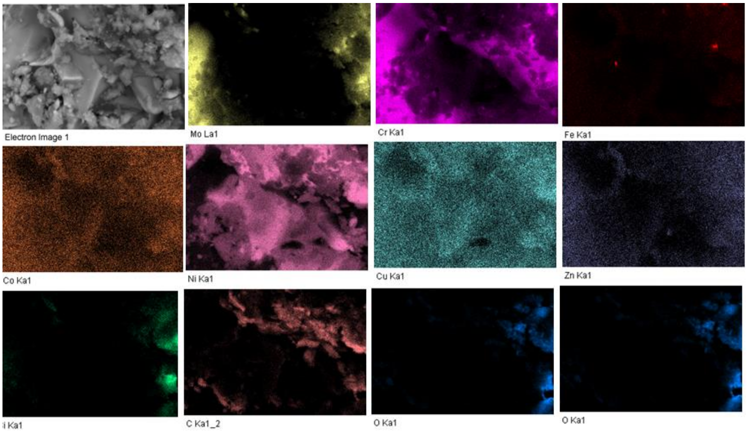

Figure 5.61: Cr, Fe, Mo, I, C Visible particles by Electroscope

Incident electron beam is fixed at defined points to scan within a special area or line, a spectrum can be obtained, from which the chemical compositions of the electron-hit point or area can be determined either qualitatively or quantitatively. Figure 5.64, 5.65 shows

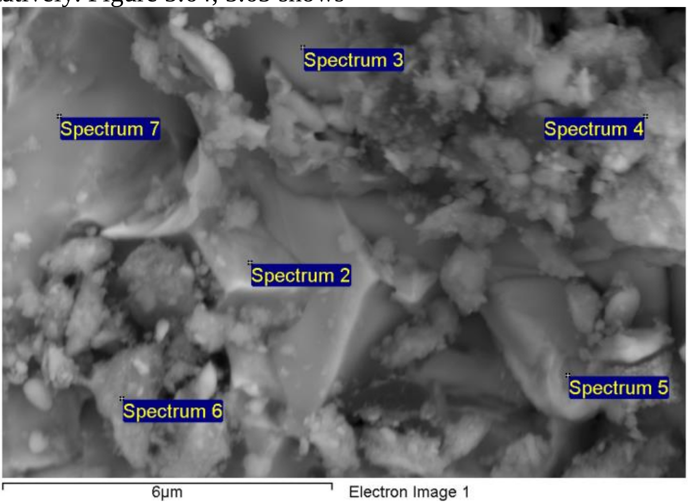

Figure 5.64: Spectrum Material analyse

Spectrum	In stats.	B	C	O	Si	Cr	Fe	Co	Ni	Cu	Zn	Mo	Total
Sum Spectrum	Yes	22.50	12.81	2.13	0.10	17.73	0.46	0.02	40.74	0.18	0.10	3.23	100.00
Spectrum 2	Yes	19.30	8.11	0.56		10.54	0.43		60.45	0.25	0.12	0.24	100.00
Spectrum 3	Yes	21.34	10.04	0.81	0.03	6.74	0.29		60.15	0.19	0.09	0.31	100.00
Spectrum 4	Yes	16.97	9.32	4.44	0.32	12.40	0.42		52.67		0.26	3.18	100.00
Spectrum 5	Yes	17.93	8.15	0.98	0.14	12.53	0.51		58.50	0.21	0.12	0.93	100.00
Spectrum 6	Yes	28.60	23.78	3.08	0.08	15.15	0.37	0.05	24.41	0.17	0.07	4.24	100.00
Spectrum 7	Yes	11.16	6.22	0.58	0.03	53.50	0.28		18.02	0.30	0.11	9.79	100.00
Max.		28.60	23.78	4.44	0.32	53.50	0.51	0.05	60.45	0.30	0.26	9.79	
Min.		11.16	6.22	0.56	0.03	6.74	0.28	0.02	18.02	0.17	0.07	0.24	

Figure 5.65: Processing option : All elements analysed (Normalised) all results in weight

Heat treated tablet & heat treated disk:

X-ray mapping is used to obtain the distributions of specific elements. In this analysis, the electron probe is scanned over a specific area and characteristics X-ray with specific energies are acquired. Figure 5.66, bellow, show X-ray mapping for particles from tribometer test under electron microscope. Particles are from tribometer test combination heat treated tablet vs heat treated disk.

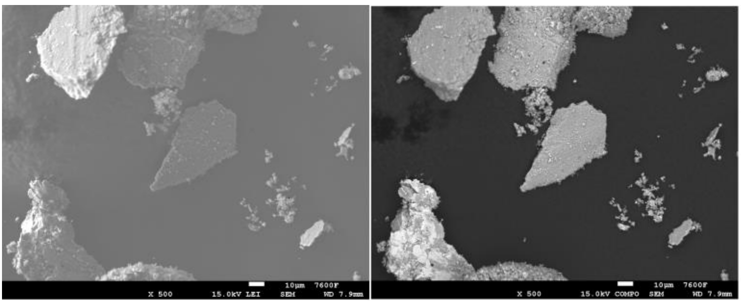

Figure 5.66: LEI/ COMPO 10μm heat treated tablet vs heat treated disk; Analysis of particles by means of three SEM modes, the SEM SE imaging mode.

Chemical Mapping by backscattered electrons:
Backscattered electrons are those scattered backwards and emitted out of the specimen. When the incident electrons are scattered in the specimen. They are sometimes called reflected electrons. Backscattered electrons carry higher energy than secondary electrons, therefore backscattered electrons bring and contain information from a relatively deep region and showing the composition of the specimen with high sensitivity.

The atomic number of the constituent atoms in the specimen is larger, which reflect larger backscattered electrons yield. Areas in backscattered electron image appear with higher brightness consist of a heavy atom. Output of this chemical mapping will be visible as image showing areas with different brightness which is suitable for observing a compositional difference.

In this dissertation experimental the scanning was done by electron microscope equipped with EDS (Energy dispersive X-ray spectroscopy), detector Oxford X-Max 50[mm^2] for chemical analysis (element mapping).

If the specimen surface has irregularity, the intensity of backscattered electrons becomes higher in the direction of specular reflection. The feature can be used to observe the topography of the surface.
Therefore, chemical analyze by using electron beam will show chemical map for materials existence in particles shape, which can move freely in testing atmosphere and surroundings.
Figures 5.67, 5.68 show visible particles by Electron microscope are the following: Chromium , Nickel, Iron, Molybdenum, Cobalt, Copper, Zinc, Silica, Boron, Carbon, and Oxygen. Existence of each material will be visible by dots or areas with higher contrast color comparing to area around. Chemical mapping will make sorting for each chemical material in separated photo and out of these photos we can clearly confirm Co or Ni particles existence.

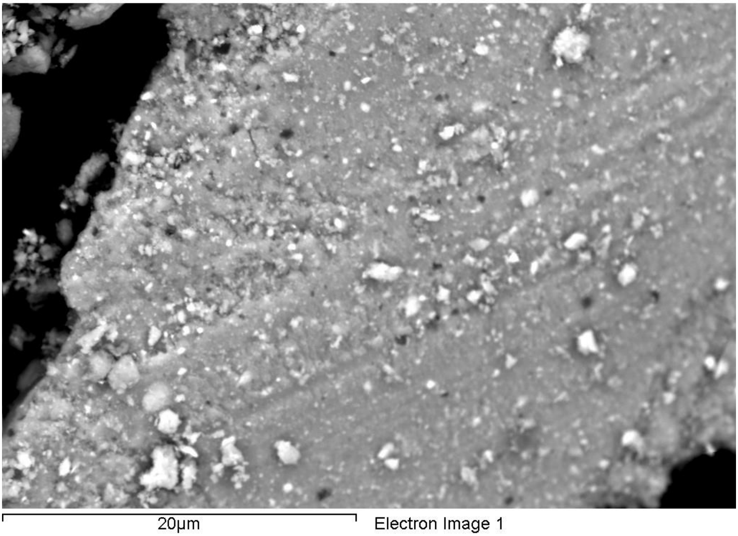

Figure 5.67: Heat treated tablet vs Heat treat disk with scale diameter 20m, Micro & Nano sized particles are visible.

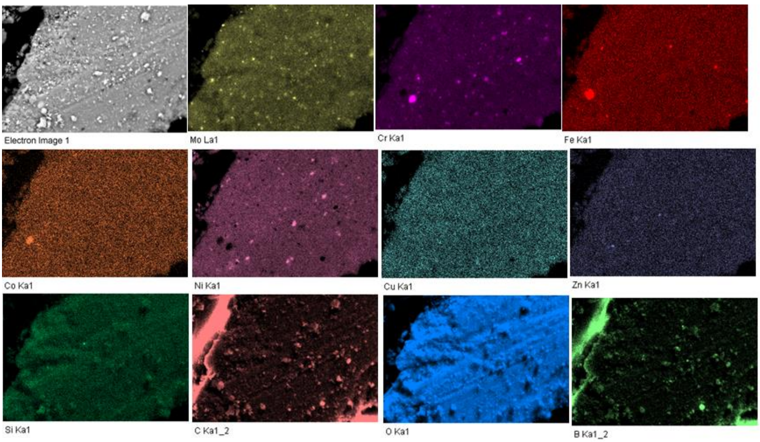

Figure 5.68: Heat treated tablet vs Heat treat disk, Chemical mapping showing MO, Cr, Fe, Co, Ni, Cu, Zn, Si, C, O, B.

4. VÝSLEDKY

The necessity to estimate risk resulted from previous studies concluded that Cobalt (Co) can be found in rocks, soil, water, plants, and animals, including people. It can harm human kind by different ways. The way how it will harm is mentioned here: eyes, skin, heart, and lungs. Exposure to cobalt may cause cancer, therefore workers may be harmed from exposure to cobalt and cobalt-containing products. The level of harm depends upon the dose, duration, and work being done [36] [A.5]

Some examples of workers at risk of being exposed to cobalt include the following:

1. Workers who work in industries processing cobalt-alloys.
2. Miners who work in the metal mining industries.
3. Workers involved in the production or use of cutting or grinding tools
4. Employees who work at nuclear or irradiation facilities

According to European chemical agency and the classification provided by companies to ECHA in REACH registrations identifies, Cobalt considered as Dangerous material and can cause serious danger on human being.

In addition to cobalt, this thesis concentrating on nickel particles existence due to face that it is part of cermets composites study case.

People are exposed to nickel and nickel compounds in many common workplaces, such as mining, welding, casting, and grinding. Occupational exposure to nickel occurs mainly through inhalation of dust particles and fumes or through skin contact, which can increase risks of lung cancer and nasal cancer.

The necessity to study this risk came from lack of knowledge and experience with this new combination of cermets (Ni625 and B4C), that is why collecting particles, produced by tribology test, for confocal microscope analyses, is necessary for this research, to distinguish micro/ nano particles existence. Electron microscope analyze was performed to confirm cobalt/ Nickel particles existence in free micro/ nano particles, which is considered as hazard, allergic particles for human being and prohibited material to be found in micro/ nano size particles [1].

Clear intersect between authors from the same field opinion and this doctoral thesis to show necessity of having risk assessment for any cobalt/ nickel usage as a risk material for any industrial usage. This idea was also supported by thesis author's published article on Metal 2021 conference [A.7].

In this thesis cermets containing B4C and Ni625 alloys as promising composite for better industrial efficiency. Target here is to show that usage of these cermets will emit particles which contain cobalt and nickel particles in nano/ micro size.

- The first main objective of this dissertation was to extend on the till now developed approaches of tribological coating and it's risk of usage in manufacturing applications.

- The risk assessment prepared by this work includes practical analysis of Inconel cermets applications in tribological testing areas.

- Further, as was seen in Section 5.2 Temperature Measurement during samples Preparation.

Welding process was continuous and no fast decreasing temperature of the work piece was obtained. Highest temperature obtained from Thermocouple point T1 and it did not increased over 900°C. Duration of time between preheating up to 300^ºC and getting back to same temperature after passing arc weld on sample, takes between 3.6-5.36 [min] depending on processing parameters, which is matching with existing research

5. ZÁVĚR

In this dissertation work, a preliminary objective was to further investigate the tribological behaviour of cermets B4C/ Ni alloys 625, as a promising composite to reduce friction, material loss and increase wear resistance. Study was fulfilled under special conditions of heat treatment and different methods of tribology testing methods were done. Further investigation behind the advantages and disadvantages of these materials on industrial tribology and related processes on the environment.

Particles analyze was done by different methods, starting with Confocal microscope as first step and electron microscope as second step. Findings from this study:

1. Confocal microscope and electron microscope were used to identify and recognize size of produced particles. Nano sized particles and micro sized particles have been observed clearly.

2. Samples with heat treatment has higher average material loss during tribology test.

3. Micro & Nano sized Cobalt particles were noticed in particles produced by tribology test (variant heat treated disk vs heat treated tablet) more than variant (heat treated disk vs non heat treated tablet).

4. Micro & Nano sized Nickel particles were noticed in particles produced by tribology test (variant heat treated disk vs heat treated tablet) more than variant (heat treated disk vs non heat treated tablet).

5. Higher care is needed here for handling discharged particles from this studied material.

6. It is important to destroy these harmful particles as preventive action by solving it into liquid or by another possible after treatment methods (Burning, Liquidation, Chemical reaction, ...), which could be part of case study future work.

Cobalt is a technically important metal, used mainly as a binder in hard-metal industry and as a constituent of many alloys. Boron Carbide and Nickel alloys compounds are used as case study in this dissertation.

Implantation and preparation of cobalt alloys has been prepared by plasma powder transferred arc welding, and cobalt compounds induced local and sometimes metastasizing sarcomas in life being. An indication of possible harmful effects of cobalt alloys or compounds in human populations has arisen from medical use, in hard-metal industries, and at cobalt production.

Unfortunately, having boron carbide and nickel alloys has a major problem, and the size of most of the investigated populations has been rather small, so none of the investigations alone gives sufficient evidence of a carcinogenic effect in humans, but taken together there is an indication of a potential risk that should be explored further [26].

From these respective results, this work may conclude the effectiveness of the approach as a means of showing risk of this material usage and possible impurities during manufacturing processes. In addition, a major component of this dissertation work was to provide mechanical properties during all tests running to highlight possible mechanical advantages by using this material in tribological applications.

Publikace související s tématem disertace

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Summary

This work conclude the effectiveness of the approach as a means of showing risk of this material usage and possible impurities during manufacturing processes of boron carbide and nickel alloys compounds, which have been prepared by plasma powder transferred arc welding and used in tribological field of applicaiotn. An indication of possible harmful effects of cobalt & nickle alloys or compounds in human populations has arisen as major problem.

In addition, a major component of this dissertation work was to provide mechanical properties during all tests running to highlight possible mechanical advantages by using this material in tribological applications.