Evaluation of Mechanical Properties of Composite Ni Base Coatings using Small Punch Test Method

Ladislav Kander [1, a], David Mikuláš [2,b] and František Kristofory [2,c]

[1]MATERIAL & METALLURGICAL RESEARCH Ltd., Pohraniční 31, 706 02, Ostrava, CZ

[2]VŠB-TU Ostrava,FS,17.listopadu,708 33, Ostrava, CZ

[a] ladislav.kander@mmvyzkum.cz,

[b]david.mikulas@vsb.cz,

[c]frantisek.kristofory@vsb.cz

Keywords: small punch test technigue, tensile strength, composite coating, nickel, electrodeposition, friction coefficient Abstract.

This paper is a study of possibilities of mechanical properties evaluation (especially tensile strength) of the electrodeposited composite coatings using small punch test method. The composite coatings on a base of Ni with contents of SiO_2, Al_2O_3, SiC, TiCN particles were electrodeposited. The layers were deposited in a wide range of current densities. The mechanical properties were obtained using very potent small punch test technique. The Small Punch (SP) miniaturised test technique is a potent method capable of providing direct values for mechanical properties of materials. The state of the art of SP technology now enables to obtain mechanical properties like yield stress, strength, elongation, transition temperature, fracture toughness, fatigue as well as creep properties of materials.

Obtained results were compared with tensile strength of pure Ni coatings and Ni plate manufactured by conventional metallurgical process.

Based on the experimental results we can conclude that small punch test method is very interesting tool for evaluation of tensile strength of composite coatings. Based on this data we can optimize electrodeposition process to develop a high tensile strength type of coating with very good wear resistance properties.

Introduction

The evaluation of mechanical properties, especially in the case of electrodeposed composite coatings, is very difficult process. The composite coatings are generally performed as thin films or layers and therefore it is practically impossible to manufacture standard test specimen for mechanical properties evaluation. Because of its high hardness there is practically impossible to cut samples from composite coating by conventional technological processes [1]. Hardness and microhardness measurements seem to be the most used (and may be only one) technique to get an idea about mechanical properties. More sophisticated approach based on evaluation of wear resistance properties takes a lot of time and is probably quit expensive.

Evaluation of mechanical properties is by definition a destructive technique, since it requires sampling material directly from the coatings. This can usually be not so easy both of the presence of internal stresses in the coating and very high hardness level.

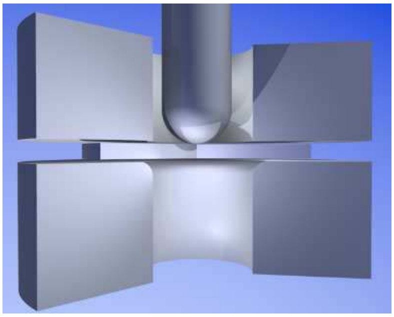
But if the sample size is so small very potent miniaturized test method can be used. For mechanical properties evaluation a small punch test technique can bee chosen.

The Small Punch (SP) miniaturised test technique is a potent method capable of providing direct values for mechanical properties of materials. The small punch technique is an almost non-destructive technique for characterizing the mechanical properties especially of service – exposed plant components. The microscopic size of the specimens allows to consider almost negligible the amount of coating that has to be sampled.

The state of the art of small punch technology now enables to obtain mechanical properties like yield stress, tensile strength, transition temperature, fatigue as well as fracture toughness properties of materials. It becomes an attractive method in lifetime assessment of power generation industry. The Vitkovice-Research and Development Ltd., has been devoted in SPT technology for 9 years and is pioneers in Czech republic in this field. Principle of this test method can be seen from Fig. 1.

The SP testing technique utilises a small disc specimen, 8 mm in diameter and 0,5 mm in thickness, clamped around its circumference and indented by a spherical punch up to failure [2,3]. The support at the border region of the disc may be loose or rigid clamping. Monotonic load vs. displacement records are used to derive estimates of tensile and fracture toughness parameters.

In this paper we can try to apply small punch test technique to direct evaluation of tensile strength of composite coatings.



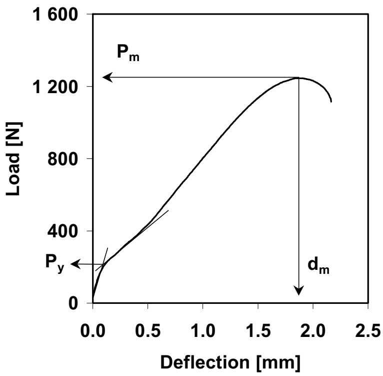


Fig.1: Principle of small punch test method and typical test record

Materials, experimental procedure and results

Electrodeposition of Ni coatings at wide range of cathodic current densities was made by standard process described for example in [4]. Electrodeposition of components coatings was made in the following way. Copper test coupons with exposed area of 9 cm^2 were used as substrates while nickel was used as the anode. The copper coupons were degreased and decontaminated. A typical nickel sulfamate electrolyte has been used for electrodeposition of coating. The composition of nickel plating bath was**:** Ni^{2+} = 1,78 mol/l , H_3BO_3= 0,49 mol/l, Br^- = 0,1 mol/l. Experiments were conducted in 0,5 liter beakers. A very small bath for components coating electrodeposition was used due to expensiveness of nanoparticles. Electrode positions were carefully maintained and solution was stirred mechanically (200 rpm and 400 rpm) to provide uniform distribution of dispersion phase in the coating.
Several types of dispersion phase have been used for electrodeposition of composite coating.

The dispersion phases:
Silicon dioxide - SiO_2 (particle size: 15 nm),Silicon carbide – SiC (particle size: 45-55 nm -spheres)
Aluminum oxide - Al_2O_3 (sigma phase, particle size: 40-47 nm),Titanium carbonitride - TiCN (avg. particle size: 50-80 nm)

Parameters of electrodeposition:
Ni coatings: temperature: 50°C, pH value: 3,9 - 4,1; cathodic current density: 1 - 7 $A.dm^{-2}$, thickness of coating: min. 0,5 mm,
Composite coatings: 50°C, pH value: 3,9 - 4,1; cathodic current density: 3 - 5 $A.dm^{-2}$, thickness of coating: min. 0,5 mm,
After each experiment the test specimens were manufactured from coupons and prepare for punch test specimens 8 mm in diameter and 0,5 mm in thickness (position 2) (from all coatings) and tensile test (position 1) specimens (from Ni coatings only) according to Fig. 2.

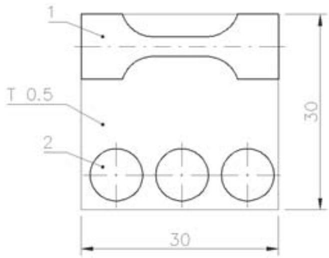


Fig.2: Cutting plan of electrodeposited coatings

Evaluation of tensile strength

<u>Ni coatings</u>

To be sure about above mentioned idea we compared the tensile strength from punch tests with tensile strength evaluated using miniaturized tensile test. We cut both the punch specimens and tensile test specimens from pure Ni coating electrodeposed at cathodic current densities between 1 to 7 $A.dm^{-2}$ as well as from Ni plate.

Because the evaluation of mechanical properties using small punch test technique is based on empirical correlation with the results of standard tensile strength and because all data up today have been collected and correlated for various qualities of steels (see Fig.3) [3], we had to obtain data for estimation of relative change between tensile test results and punch tests for Ni coating and Ni plate.

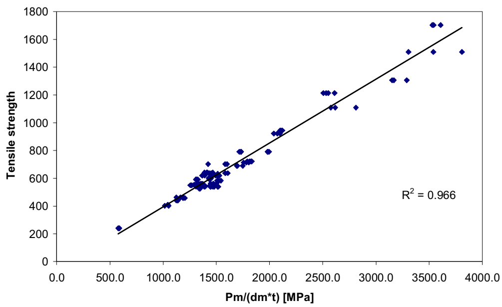


Fig. 3: Small punch test correlation for tensile strength (steels) [2]

So we test the specimens using punch test and tensile test and compare the results of tensile strength to estimate relative tensile strength shift due the change of friction ratio from steel specimen – steel punch to Ni sample – steel punch.

In the Fig. 4 is presented comparison of tensile strength evaluation using both methods on Ni coatings as well as Ni plate.

From the Fig 4 can be seen a difference between the results from tensile tests and average value from three punch tests. This difference probably comes from friction coefficient shift. The relation between load and membrane stress in the case of punch test is described mostly by equation 1-3 [5-7].

$$\frac{F_{SP}}{\sigma} = \left[2 \cdot \pi \cdot t \cdot \left(R + \frac{t}{2} . \sin\phi \right) \cdot \sin\phi \right] \quad (1)$$

$$\frac{F_{SP}}{\sigma} = \frac{2 \cdot \pi \cdot t \cdot r \cdot \tan\Theta}{\sqrt{1 + \tan^2\Theta}} \quad (2)$$

$$\frac{F_{SP}}{\sigma} = 2 \cdot \pi \cdot R \cdot \sin^2\Theta \cdot t \cdot \left(\frac{1 + \cos\phi}{1 + \cos\Theta} \right)^2 \quad (3)$$

The symbols in equations correspond to the Fig. 5.

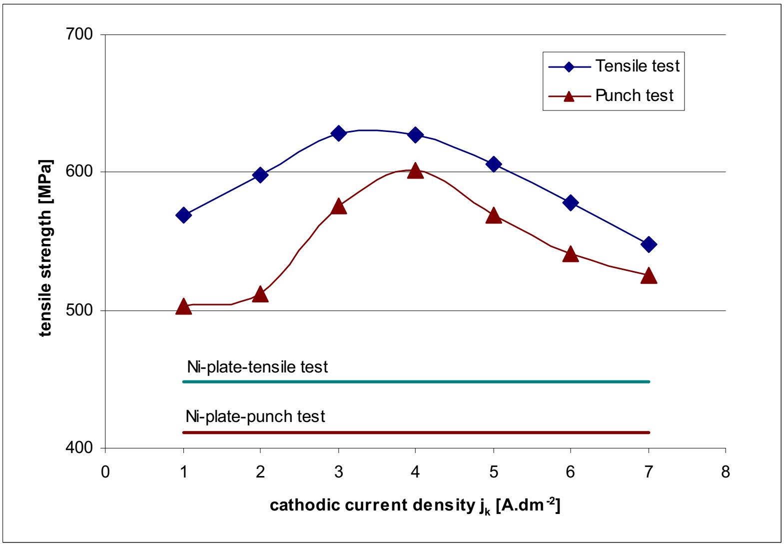


Fig. 4: Effect of current density on tensile strength Ni coating evaluated both tensile test and punch test (compared to Ni plate)

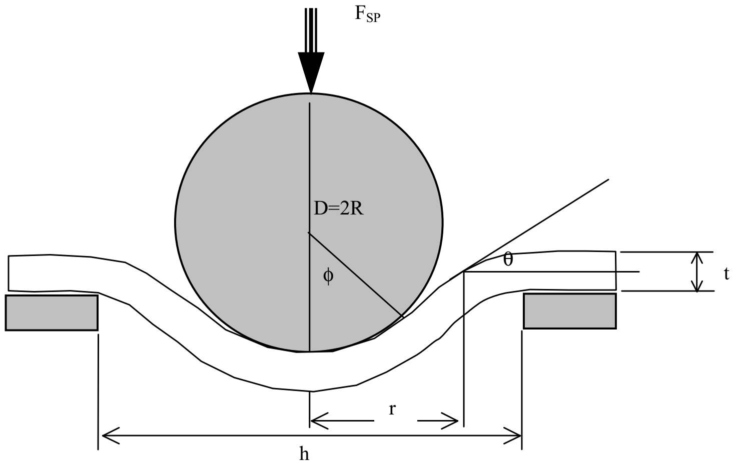


Fig.5: Geometric condition during small punch test

From all three equations (1-3) is very clear that the stress depends on geometrical conditions (angles ϕ and θ) then on friction coefficient. Based on above mentioned analysis we can conclude that for correct estimation of correlation between punch test and tensile test results is very important to know right value of friction coefficient between specimen and ball.

After recalculation the results from Fig. 4 to correct value of friction coefficient [8] we obtained the results summarised in Fig. 6.

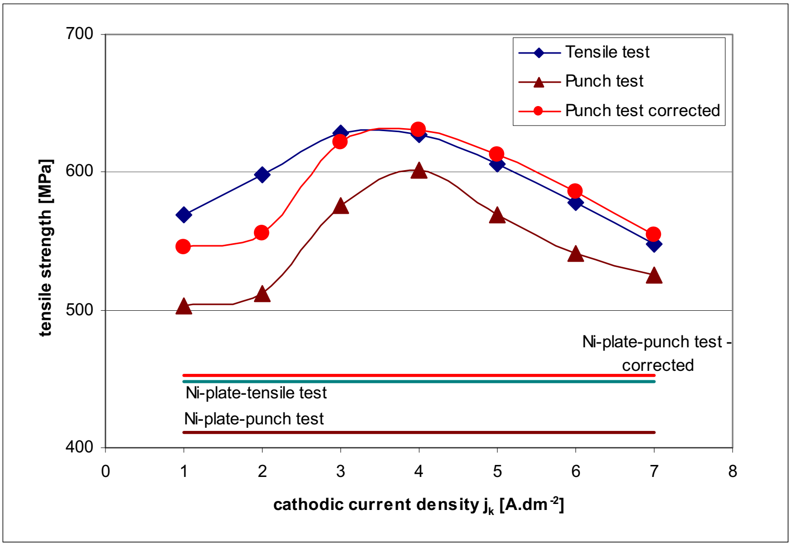


Fig.6: Results of punch tests after friction coefficient correction

Composite coatings

Mechanical properties, especially tensile strength, of the composite coatings under investigation were evaluated only by small punch test technique. From each coupon (30x30 mm) were cut the punch test specimens (see Fig.2).

The punch test were performed on INOVA TSM 10 kN servomechanical testing machine at constant crosshead speed of 0,2 mm/min using disc specimens 8 mm in diameter and 0,5 mm in thickness.

Load versus displacement was recorded to evaluate maximum force and maximum deflection (see Fig 1). From these values tensile strength was calculated using correlation equations developed in Vítkovice – Research and Development, Ltd. (see Fig. 3). All data were corrected on friction in the same way as Ni coating results.

The results obtained on various types of composite coatings electrodeposed at different hydrodynamic conditions are summarized in Table 1.

Table 1: Results of tensile strength of composite coatings

Coating	Stirring revolution per minute [min^{-1}]	Tensile strength evaluated using small punch tests and corrected [MPa]
Ni+SiC	200	565
Ni+SiC	400	760
$Ni+SiO_2$	200	650
$Ni+SiO_2$	400	695
$Ni+Al_2O_3$	200	980
$Ni+Al_2O_3$	400	1040
Ni+TiCN	200	1350
Ni+TiCN	400	1600

Discussion

Ni coatings and Ni plate

From the experimental results presented in Fig. 4 can be seen that the results of tensile strength of Ni coating evaluated using punch tests are underestimated. Average difference between tensile strength estimated using tensile test and punch test is about 35 MPa in the range of cathodic current densities from 3 to 7 $A.dm^{-2}$. If the current density is lower (from 1 to 3 $A.dm^{-2}$), the average difference increases to 68 MPa. This increasing can be explained either by deposition of impurities (such as Fe) in the coating which is typical at low cathodic current densities or hydrogen embrittlement. Impurities or hydrogen can affect the tensile strength in small tested volume (punch test) dramatically. Detail study of grain boundaries of such deposited coating will be needed to confirm above mentioned idea.

On the other hand in the range of cathodic current densities from 3 to 7 $A.dm^{-2}$ the average difference of both test method is only 35 MPa, this present deviation about 10 %. This precision is quit well even before any correction, but from physical point of view this results could not be taken as satisfactory.

After taking the friction effect into account the difference between tensile tests and punch test are very smaller and correspond near 4 MPa. For example the tensile strength of Ni plate evaluated by tensile test was found to be 448 MPa, corrected value of tensile strength from small punch test was found to be 452 MPa. This fact can be seen in the Fig. 6.

We must also notice that tensile strength values after correction are little overestimated (see Fig. 6) both for Ni coating and for Ni plate. This overestimating can be probably caused by uncertainty in friction coefficient estimation because of lack of experimental results on this field. Generally in the Fig. 6 there is a very good agreement in the experimental results from tensile tests and corrected tensile strength evaluated using punch tests, especially if the current density is above 3 $A.dm^{-2}$.

Effect of friction can be expressed without any difficult calculation using easy correction between real tensile strength and tensile strength evaluated by punch tests in the form of equation (4).

$$\mathbf{R_{m\,REAL} = R_{m\,Punch\,Test} + 35\,MPa} \qquad (4)$$

This easy correction is precise enough to predict the tensile strength of Ni base coatings as well as for Ni plate.

It is clear that small punch test method is very potent test method capable to evaluate mechanical properties of electrodeposed coatings. The proposed simple correction in equation (4) based on friction coefficient calculation seems to be very useful tool to evaluation real tensile strength of Ni coatings as well as Ni plate. Based on this data we can optimize electrodeposition process to develop a high tensile strength type of coatings with very good wear resistance properties.

Composite coatings

Mechanical properties, especially tensile strength, of the composite coatings under investigation were evaluated only by small punch test technique. The results are summarized in Table 1. In right column in the Table 1 there are plotted values of tensile strength of various types of composite coatings corrected on real value using equation 4.

We assume that the effect of particles on friction coefficient is negligible and friction does not change in comparison with Ni coatings or Ni plate significantly. Even if this assumption is not correct, relative shift of tensile strength is correct (for example for evaluation of effect of hydrodynamic condition on tensile strength).

Of course final macroscopic tensile strength (or any mechanical properties) in the case of composite coatings is function of size and distribution of particles in the coating as well as hydro and electrodynamical conditions during the process of electrodeposition.

Generally the tensile strength of composite coating with above mentioned particles type increase compared to Ni coating. However from the Table 1 can be seen that tensile strength of electrodeposed composite coatings is a function of dynamical conditions of electroplating process. The hydrodynamic conditions, especially stirring revolution, affect mechanical properties significantly in all composite coatings under investigation. If the revolutions per minute are increased from the value 200 per minute to 400 per minute the significant change in strength of coating under investigation can be observed. This can be probably explained by better distribution of particles in the matrix. The highest level of tensile strength was observed in coating with TiCN particles, tensile strength level was evaluated between 1350 and 1600 MPa with respect to hydrodynamic conditions.

On the other hand coating with SiC particles at low stirring revolution (200 per minute) show decreasing of tensile strength compare to Ni coating. Further fractographical and TEM studies are needed to explain this effect.

Summary

In this work several tensile strength data sets have been analyzed. Based on the preliminary evaluation of the experimental results the following conclusion can be drawn:

- small punch test method can be use to evaluation of tensile strength of Ni base composite coatings
- the results of tensile strength for Ni coating and Ni plate based only on current correlation between standard tensile tests and small punch test are underestimated due to change of friction coefficient between specimen and punch, after recalculation with the right value of friction correspond to material to be tested correct value of tensile strength can be obtained.
- simple correction for Ni base coating was proposed to provide useful tool for direct estimation of tensile strength for Ni base coatings. Very good agreement between experimental results was found using this correction.
- type, size of particle and hydrodynamic conditions, especially stirring revolution, affect mechanical properties significantly in all composite coatings under investigation.
- the highest level of tensile strength was observed in composite coating with TiCN particles, tensile strength level was evaluated between 1350 and 1600 MPa with respect to hydrodynamic conditions.
- pioneer study in the field of evaluation of mechanical properties of composite coatings using small punch test technique was presented in this paper. New approach of evaluation of mechanical properties of composite coatings has been introduced and its descriptive potential with respect to tensile strength estimated using tensile test has been evaluated.

Acknowledgements

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