

SURFACE MODIFICATIONS OF MARAGING STEELS USED IN THE MANUFACTURE OF MOULDS AND DIES

MODIFIKACIJA POVRŠINE JEKLA MARAGING IN UPORABA PRI IZDELAVI KOKIL IN UTOPOV

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Low-carbon, high-alloy, precipitation hardening MARAGING steels have been developed in the early sixties of the last century as a high strength structural materials for application in aeronautical and missile engineering. Due to their excellent properties, such as: high hardenability, good toughness, and high resistance to thermal fatigue, and due to simple heat treatment (without protective atmosphere) with very small distortions, MARAGING steels are successfully used for the fabrication of moulds and dies. The main drawback of these steels is their relatively low wear resistance, particularly if the die is subjected to extensive wear during service. The paper presents an overview of existing MARAGING steels used in mould manufacturing and gives their general properties. Also, the results of the applied modification and coating processes are presented with a special focus on the increase in wear resistance.

Key words: MARAGING steels, thermo-chemical treatment, PVD coating, wear resistance

Maloogljiva, visokolegirana in izločevalno utrjena jekla MARAGING so bila razvita v zgodnjih šestdesetih letih prejšnjega stoletja kot zlitine z visoko trdnostjo za uporabo v letalski in raketni tehniki. Zaradi izvrstnih lastnosti, kot so velika kaljivost, dobra žilavost, velika odpornost proti toplotni utrujenosti in enostavna toplotna obdelava (brez varovalne atmosfere) z majhnim krivljenjem, omogočajo učinkovito uporabo teh jekel tudi za izdelavo kokil in utopov. Njihova pomanjkljivost je majhna odpornost proti obrabi, še posebej, če je utopno orodje izpostavljeno veliki obrabi. V članku je pregled jekel vrst MARAGING, ki se uporabljajo pri izdelavi utopov, in njihovih lastnosti. Predstavljeni so tudi rezultati uporabljenih metod za obdelavo in prekritje površine s poudarkom na povečani odpornosti proti obrabi.

Ključne besede: jekla MARAGING, termo-kemična obdelava, PVD-prekritja, odpornost proti obrabi

1 INTRODUCTION

Low-carbon, high alloy MARAGING steels belong to the Fe-Ni-Co alloying system, using Mo, Ti, and Al as alloying elements. These steels were developed in the early sixties of the last century as high strength structural materials intended for application in aeronautical and aerospace engineering (e.g. for propeller drive shafts, pilot seat frames, liquid fuel tanks, armour plates, etc.). By modifying their composition with the addition of Cr (9–3 %), Fe-Ni-Cr or Fe-Ni-Co-Cr alloying systems were created, MARAGING steels belonging to these systems are suitable for the application in highly corrosive environments². Later on, MARAGING steels started to be used for the manufacturing of tools, where they are superior to other tool materials due to their superior properties¹⁻³:

- High toughness and high fracture toughness combined with very high strength;
- High resistance to thermal fatigue;
- Protective atmosphere in heat treatment is not required, i.e. in solution annealing and precipitation hardening – aging, as there is no risk of decarburization and oxidation of the surface.
- Hardenability, even with the largest tool sizes, is achieved by slow cooling from the temperature of

solution annealing; therefore, the risks of distortions and the occurrence of cracks caused by the difference in temperatures in the tool cross-section are significantly reduced.

- Good weldability;
- Good electro-erosion machinability;
- Good chip machinability and/or cold deformability after quenching enable the tool manufacturing to the end dimensions before the final heat treatment (aging) is completed.
- In solution annealing and in cooling, the expected shortening is approximately 0.1 %, and in the final heat treatment (aging), there are practically no deformations; therefore, these heat treatment processes can be considered as processes causing no distortion.
- Compared to the conventional heat treatment of tool steels, the heat treatment of MARAGING steels (solution annealing and aging) is much simpler.

The main disadvantage of MARAGING steels compared to high alloy tool steels is their relatively low hardness ($HRC = 50-57$ at the most), and consequently the insufficient resistance to wear. Due to their lower hardness, MARAGING steels are not suitable for the manufacture of cutting tools, but are suitable for the manufacture of moulds and dies (for die-casting and for

polymer processing), of forming tools, etc.^{1,2}. A relatively high price of MARAGING steels (they are several times more expensive than the high alloy tool steels produced by standard methods) cannot be taken as a major drawback in modern machine tool industry since tool steels produced by powder metal forming have a similar price.

In references³⁻⁵, the application of nitriding and/or nitrocarburising is usually recommended to improve the wear resistance. The investigation of the application of other thermo-chemical treatments (carburizing, boriding) and of vapour deposition coating processes⁶⁻¹⁰ seems to be a natural choice. Results found in literature and results of our research on the possibility of improving the wear resistance of MARAGING steels are presented in this work.

2 PROPERTIES OF MARAGING TOOL STEELS

Iron-nickel phase diagram and the influence of the content of nickel on the hardness in the solution-annealed steels (**Figure 1**) can contribute to the understanding of the hardening mechanism of MARAGING steels. **Figure 1 a**, shows that the alloy with approximately 18 % Ni will be in the single-phase austenite area above the temperature of 650 °C (generally the temperature of austenization-homogenization is approximately of 820 °C). With this content of nickel, the alloy exhibits the highest strength after cooling (**Figure 1b**). As the solution annealed MARAGING steel is cooled from the temperature of annealing to approximately 250 °C (slow cooling in the air is sufficient), the low-carbon

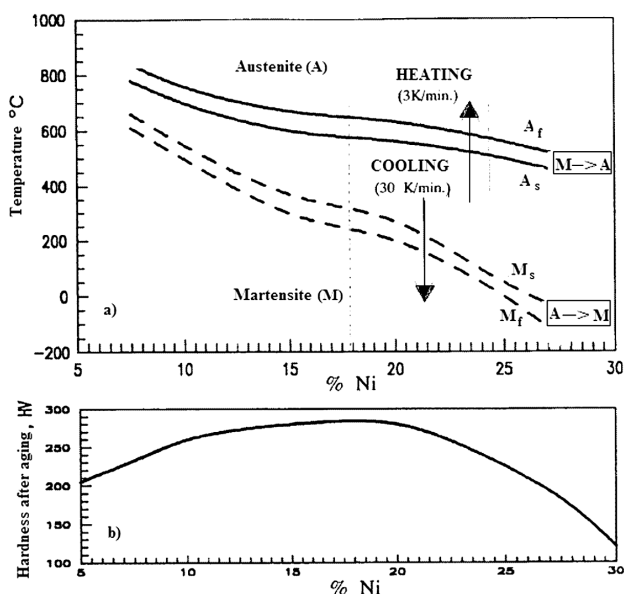


Figure 1: a) Real phase diagram Fe-Ni¹¹, b) Quenching hardness of Fe-Ni alloys in dependence of the percentage of nickel (with the addition of Co, Mo, Ti, and Al)¹²

Slika 1: a) Realni fazni diagram Fe-Ni¹¹, b) Kalilna trdota zlitin Fe-Ni v odvisnosti od vsebnosti niklja (z dodatkom Co, Mo, Ti in Al)¹²

nickel-martensite, which is actually an oversaturated solution of Co, Mo, Ti and Al in the alloy Fe-Ni(Co), starts to form. The transformation of austenite into nickel-martensite is completed at a temperature of approximately 200 °C; and at this temperature, the microstructure should be without residual austenite. Nickel-martensite (with a hardness of approx. HV 300) can be easily machined by the chips removal and by deformation machining and can even be welded, if necessary.

The solution annealing of MARAGING steels is carried out in steel mills, and the manufacturer of a machine part or a tool carries out only the final heat treatment (aging) after the machining to the specified dimensions. The process of aging is performed in furnaces without protective atmosphere and at relatively low temperatures (approx. 500 °C). In the slow cooling process that follows, small distortions occur. In the age hardening process, a large number of finely dispersed precipitates of intermetallic phases occur (e.g. Ni₃Al, Ni₃Ti, Fe₂Mo, FeCr, and Fe₇Mo₆) in nickel martensite, which results in an increase in steel hardness from HV = 280–380 to HV = 500–650. Also, the tensile strength of the steel is increased to 4200 N/mm², and deformability and fracture toughness are rather good¹⁻⁴. **Figure 2** gives a flow diagram of the heat treatment process of MARAGING steels.

Table 1 lists several grades of MARAGING tool steels divided into three groups according to their resistance to elevated temperatures and corrosion²:

- steels suitable for hot working at temperatures of up to 425 °C: X 3 NiCoMo 18 8 5, X 3 NiCoMo 18 9 5, X 2 NiCoMoTi 18 12 4
- steel suitable for hot working at temperatures of up to 600 °C: X 2 NiCoMo 12 8 8
- steels with superior corrosion stability: X 1 CrNiCoMo 9 10 3, X 1 CrNiCoMo 13 8 5, X 2 CrNiCoMo 12 8 5

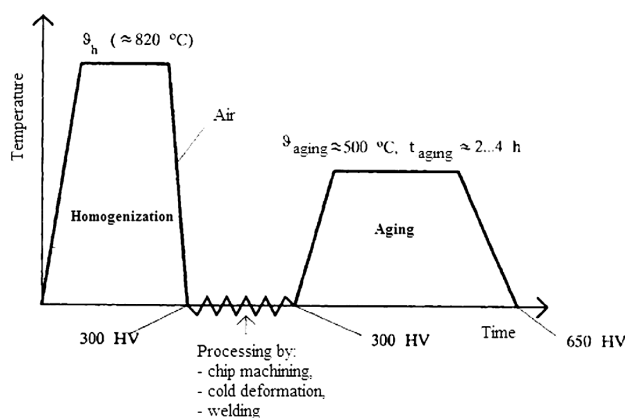


Figure 2: A flow diagram of the heat treatment process of MARAGING steels²

Slika 2: Diagram poteka procesa toplotne obdelave jekel MARAGING jekel²

Table 1: MARAGING tool steels ²

Tabela 1: Jekla MARAGING ²

Resistance to temperature / corrosion	Steel grade	Heat treatment		Mechanical properties									
		Homogenization temperature, °C	Aging temperature, °C / Aging time, h	after solution annealing (homogenization)					after aging				
				R_m /MPa	$R_{p0.2}$ /MPa	max. HV	min. A ₅ /%	min. KV _{20°C} /J	R_m /MPa	$R_{p0.2}$ /MPa	max. HV	min. A ₅ /%	min. KV _{20°C}
<425 °C	X3 NiCoMo 18 8 5	820	480 / 3	1130	830	350	15	60	1920	1720	500	8	20
	X3 NiCoMo 18 9 5	820	480 / 3	1130	830	350	15		1960	1910	570	7	30
	X2 NiCoMoTi 18 12 4	820	500 / 6	1130	830	350	15		2350	2260	590	6	10
<600 °C	X2 NiCoMoTi 12 8 8	900	550 / 2						1980	1800	560		
Corrosion resistance	X1 CrNiCoMo 9 10 3	835	480 / 6						1500	1400		12	60
	X1 CrNiCoMo 13 8 5	820	480 / 6						1700	1500		11	50
	X2 CrNiCoMo 12 8 5	880	480 / 6	1000	700				1870	1650		10	40

Table 2: Chemical composition of MARAGING steel 14 10 5

Tabela 2: Kemična sestava jekla MARAGING 14 10 5

w/%															
C	Si	Mn	P	S	Cr	Mo	Ni	V	Al	Cu	Ti	Nb	N	B	Co
0.01	0.88	0.087	0.003	0.002	0.11	4.81	13.60	0.024	0.14	0.08	0.17	0.10	0.013	0.002	9.50

MARAGING steels suitable for working at temperatures of up to 425 °C have a rather low resistance to high temperatures since austenite, which decreases the hardness and strength in operating conditions, is recreated already at the temperature of 500 °C ¹³. Therefore, these steels are used for tools used in cold and lower temperature operating conditions, such as: ²:

- moulds for polymer processing
- dies for the die casting of silumin or zinc alloys
- punches for the extrusion of lead cable sheaths
- tools for compression-extrusion of Al-alloys
- matrices and punches for the cold forging of bodies and heads of bolts.

The MARAGING steel suitable for hot working at temperatures of up to 600 °C (X 2 NiCoMo 12 8 8) has a lower content of Ni than the steels with 18 % Ni. Therefore, it has to be solution annealed at a temperatures of approximately 900 °C (**Figure 1**) and also precipitation hardened (aged) at temperatures of up to 625 °C. The increased content of Mo (from 5 % to 8 %) has also a beneficial influence on the increase in hardness during the aging process by additional precipitation of Ni₃Mo. Due to the improved resistance to thermal fatigue, this steel grade can be recommended for the manufacture of mould components for the die casting of aluminium alloys ².

MARAGING steels with improved corrosion stability have a lower content of nickel (from 8 % to 10 %) and cobalt Co (from 3 % to 5%) and contain between 9 % to 13 % of chromium (as an active corrosion resistant ingredient, chrome contributes to the improved corrosion

resistance). The addition of chromium decreases the hardness, as well as the strength (to approximately 1600 N/mm²) and the yield stress (to approx. 1500 N/mm²). It decreases also, the temperatures of the start (M_s) and the finish (M_f) of the formation of nickel-martensite. Therefore, with these steels, retained austenite may occur after quenching and a need for deep cooling may arise. MARAGING steels high-alloyed by chrome can be recommended for the manufacture of mould components for the processing of highly corrosive polymers.

3 IMPROVED WEAR RESISTANCE

In reference, results of research into the feasibility of the application of various surface treatment procedures (nitriding, carburizing, boriding, and PVD coating) in order to improve the wear resistance of MARAGING steels are found ^{3,6,7,9,10,11,12,13}. Our investigations ^{6,9} on the MARAGING steel 14 10 5 (**Table 2**) included the following surface treatment procedures: nitriding in Tenifer salt bath, nitriding in plasma gases, as well as carburizing and boriding according to the parameters listed in Table III. In addition, a possible application of the duplex treatment of plasma nitriding and of physical vapour deposition (PVD) procedure has been investigated also. In **Table 3** the performed researches are summarised and all heat treatment parameters applied are listed.

The effect of processes of modification and coating on the properties of MARAGING steel 14 10 5 has been estimated with microstructure, hardness distribution on

Table 3: Heat treatment of test samples made of MARAGING steel 14 10 5

Tabela 3: Toplotna obdelava preizkušancev iz jekla MARAGING 14 10 5

Heat treatment parameters	
Solution annealed at 820 °C/1h/air	Aged at 500 °C/4h
	Nitrocarburized by the TENIFER procedure at 580 °C/4h
	Ion-nitrided at 500 °C/72h
	Ion-nitrided at 500 °C/20h
	Ion-nitrided at 500 °C/20h + PVD coated at 450 °C/70 min
	Carburized in the Degussa KG 6 granulate at 900 °C/4h + aged at 500 °C/4h
	Borided in the EKABOR 2 (Degussa) powder at 900 °C/4h + aged at 500 °C/4h

the cross-section and tribological tests (abrasive, erosive and adhesive wear). **Figures 3 to 8** present the results of these tests. The testing of resistance to abrasive wear was carried out by the dry sand/rubber wheel tests (Ottawa 50/70 quartz sand) at a compressive force of 45 N (**Figure 4**). The same sand type was used in the tests of erosive wear with sand grains colliding with the test sample surface at an angle of incidence of 90° (**Figure 6**). Tests of abrasive wear were carried out by a friction ring made of hardened steel at the compressive force of 100 N. During testing, values of friction coefficient were determined (**Figure 8**). Details on the conducted tests are given in ^{9,10}.

3.1 Carburization of MARAGING steels

The carburization of MARAGING steels was carried out at the temperatures of solution annealing. After the cooling process, high-carbon martensite with increased hardness (and a probable presence of residual/retained austenite) is obtained in the boundary layer. The total depth of carburized layer on the MARAGING steel 14

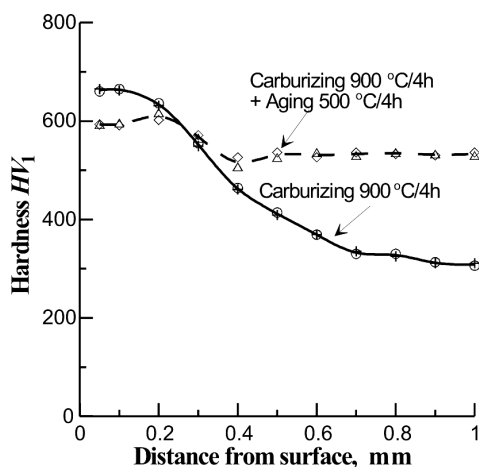


Figure 3: Hardness distribution in the edge layer of carburized test samples made of MARAGING steel 14-10-5

Slika 3: Porazdelitev trdote ob površini naoogljičene plasti pri preizkušancu iz jekla MARAGING 14 10 5

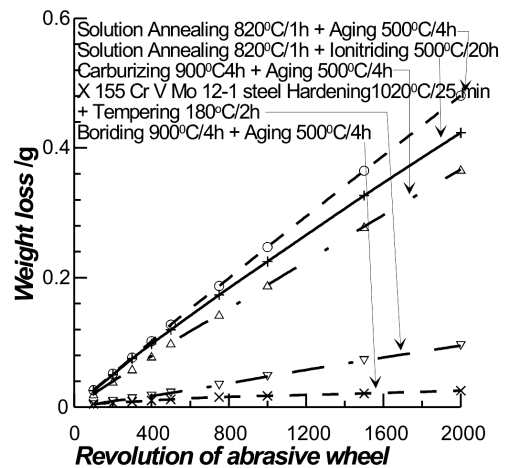


Figure 4: The abrasive wear mass loss of test samples made of MARAGING steels 14-10-5 with and without surface modifications compared with the wear of samples made of high alloy tool steel X155CrVMo12 1 ^{9,10}

Slika 4: Izguba mase zaradi abrazivne obrabe preizkušancev iz jekla MARAGING 14 10 5 z modifikacijo površine in brez nje v primerjavi s preizkušanci iz močno legiranega orodnega jekla X155CrVMo 12 1 ^{9,10}

10 5 was approx. 0.8 mm. The subsequent aging improved the core hardness, and reduced the hardness of the carburized layer (**Figure 3**). The carburization of MARAGING steels improved their resistance to abrasive wear compared with the solution annealed and aged condition or with that of solution annealed and nitrided (**Figure 4**). The carburization of MARAGING steels also has some adverse effects. The formation of a hard martensite layer (with hardness of approx. HV 700) is accompanied with additional distortions and by a more difficult chip machining. Thus, one of major advantages of MARAGING steels, i.e. the heat treatment without distortions, is lost.

3.2 Boriding of MARAGING steels

The boriding of MARAGING steel 14 10 5 by using the standard means and parameters results in a boride

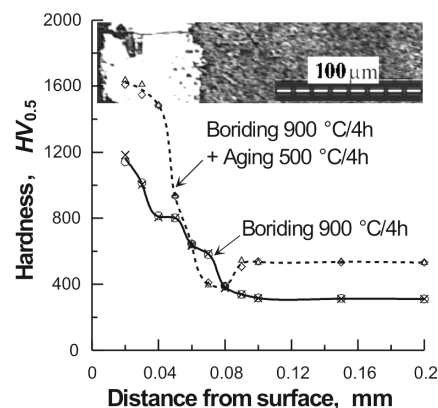


Figure 5: The microstructure and hardness distribution in the edge layer of borided test samples made of MARAGING steel 14-10-5 ¹⁰

Slika 5: Mikrostruktura in porazdelitev trdote v robni plasti boriranih preizkušancev iz jekla MARAGING 14 10 5 ¹⁰

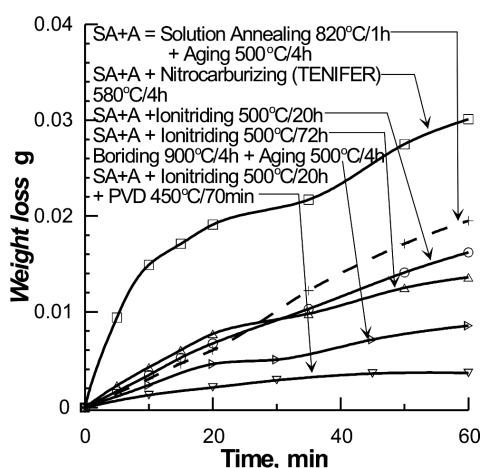


Figure 6: Mass loss in erosive wear tests carried out on test samples made of MARAGING steel 14-10-5 with and without surface modifications¹⁰

Slika 6: Izguba mase pri preizkusih erozivne obrabe preizkušancev iz jekla MARAGING 14 10 5 z modifikacijo površine in brez nje¹⁰

layer with a thickness of approx. 50 μm and with high hardness (above $HV_{0.5}$ 1000) (Figure 5). This layer showed a high resistance to erosive wear and an improved resistance to abrasive wear, even higher than that of highly wear resistant ledeburite steel for cold working (Figure 4 and 6). Along with these obvious advantages, one should also notice the problems in the application of the boriding of MARAGING steels, such as the occurrence of a soft zone below the boride layer, the presence of transverse cracks in the boride layer, and the risk of exceeding dimensional tolerances. Due to the achieved high hardness after boriding (prior to aging), it is not possible to correct dimensions and thus the property of "distortionless" heat treatment is lost.

3.3 Nitriding of MARAGING steels with and without a subsequent PVD coating procedure

An increased wear resistance of MARAGING steels can be successfully obtained by plasma nitriding^{6,9,13,14} at temperatures of approximately 500 °C, which are in the range of aging temperatures of these steels. The application of solution annealing and plasma nitriding results in an improved resistance to abrasive and erosive wear compared with the application of standard solution annealing and aging (Figure 4 and 6). Also, all the other advantages of the application of MARAGING steels pointed out before are retained. Research results reported in¹³ show that the corrosion resistance of MARAGING steels exposed to salt mist (salt-spray test with exposure of the test sample to the aqueous solution of sodium chloride at a concentration of 5 % NaCl) is increased by three times if plasma nitriding is applied compared to the solution annealed and aged condition. This result is expected in all the cases in which a zone of compounds (Fe nitrides) is formed on the MARAGING steel surface. Here, the formation of a single-phase layer of Fe_4N

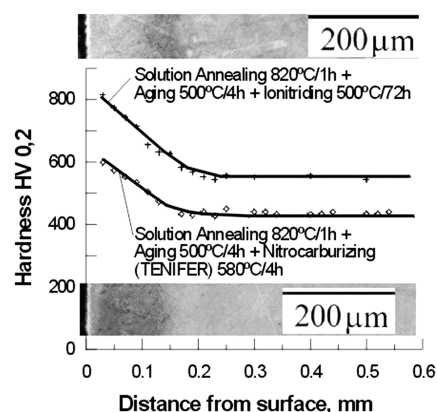


Figure 7: Microstructure and hardness distribution in the edge layer of test samples of MARAGING steel 14-10-5, nitrided in plasma and nitrocarburized in the TENIFER salt bath¹⁰

Slika 7: Mikrostruktura in porazdelitev trdote v robni plasti preizkušancev iz jekla MARAGING 14 10 5, ki so bili nitrirani v plazmi in karbonitrirani v solni kopeli TENIFER¹⁰

nitrides is recommended since, according to¹⁴, these layers exhibit a good combination of high hardness and ductility.

The application of nitriding and nitrocarburizing in gases, as well as nitrocarburizing in the Tenifer salt bath results in the overaging of the core due to a higher temperature of the treatment (550–600 °C). This phenomenon is reflected in hardness decrease of the base metal and in the hardness of the nitrided layer (Figure 7). The overaging of the core can have an influence on the decrease in the resistance to abrasive and erosive wear (Figure 6)^{9,10}.

The duplex treatment of plasma nitriding and of the physical vapour deposition (PVD) procedure seems to be of a particular interest regarding the improvement in wear resistance. In the case of the tested MARAGING steel 14 10 5, the thickness of the PVD TiN coating was 1.8 μm (Figure 8). In the plasma nitriding treatment it is possible to vary the parameters of the procedure in order

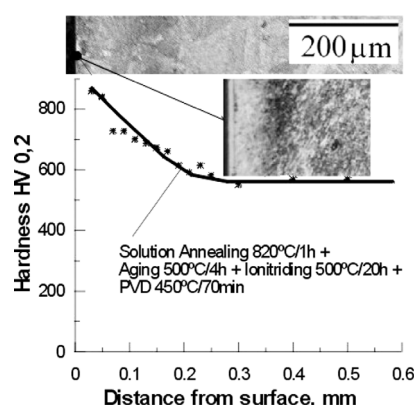


Figure 8: Microstructure and hardness distribution in the edge layer of test samples made of MARAGING steel 14-10-5, nitrided in plasma and coated by a PVD TiN coating¹⁰

Slika 8: Mikrostruktura in porazdelitev trdote v robni plasti preizkušancev iz jekla MARAGING 14 10 5, ki so bili nitrirani v plazmi in pokriti s plastjo PVD TiN¹⁰

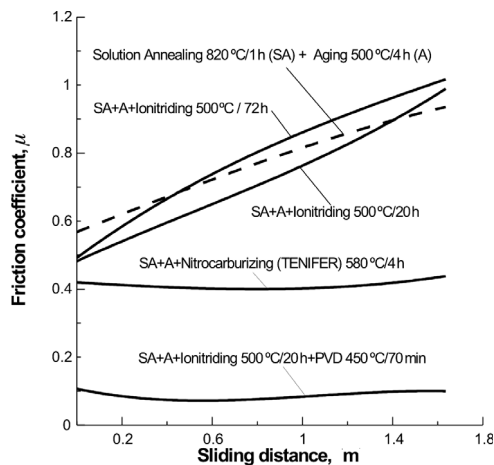


Figure 9: Friction coefficient determined in the adhesive wear testing of test samples made of MARAGING steel 14-10-5 with and without modifications of the surface ¹⁰

Slika 9: Koeficient trenja pri preizkusu adhezivne obrabe preizkušancev iz jekla MARAGING 14 10 5 z modifikacijo površine in brez nje ¹⁰

to obtain a boundary layer with or without a zone of compounds, and the PVD treatment produces a hard coating with the properties of a high resistance to abrasive, erosive and adhesive wear, together with an extremely low friction coefficient (**Figure 4 and 9**).

3.4 Repair of worn tools from MARAGING steels

Low carbon MARAGING steels can be welded, which is a good basis for the repair of worn tool surfaces and those with thermal fatigue cracks. A description of a successful welding procedure and the closure of surface microcracks in the MARAGING steel X 2 NiCoMoTi 12 8 8 with laser remelting (using a diode laser) without using a filler metal is given in ¹⁵. Diode lasers with beam power of 1 kW to 2 kW are compact and easily controlled sources of light beams suitable for the repair and maintenance of tool surfaces, especially of large and expensive tools, such as moulds for die casting, dies, tools for pressing polymers, etc. For each repair case and type of MARAGING steel, one has to determine appropriate operating parameters, such as the power output and shift of the laser beam and its focusing points, the depth of remelting, and other parameters.

Diode lasers can find their application not only in tool repair procedures for remelting tool surfaces, but also in the manufacture of tools made of MARAGING steels in order to improve the wear resistance of the surface by applying coating and alloying procedures.

4 CONCLUSION

Good mechanical and technological properties and the heat treatment with almost no distortions make MARAGING tool steels suitable for the manufacture of moulds used in polymer processing, of dies for die-

casting, drop-hammer dies, punches, matrices, and other non-cutting tools. In addition to the basic group of MARAGING steels with 18 % Ni, two special groups of steels resistant to temperatures of up to 600 °C and those used for tools exposed to corrosion have been developed.

In the investigation, it is confirmed that the rather poor wear resistance of MARAGING steels can be improved with thermo-chemical heat treatments, as nitriding, nitrocarburizing, boriding and carburizing.

The carburizing process improve the resistance to abrasion, however it cannot be recommended as a heat treatment process for the surface modification of these steels because the risk of exceeding the required dimensional tolerances and of shape changes (similarly as in the case of boriding) and the chip machining to be conducted prior to aging is made more difficult (thus, a major advantage has been lost).

The process of boriding significantly improves the resistance to abrasive and erosive wear, but transverse cracks occurring in the boride layer, a change in dimensions and the loss of "distortionless" heat treatment make this process, for the time being, unsuitable for the manufacture of tools.

Nitrocarburizing in gases and in salt baths produces good-quality layers, but since the process is carried out at very high temperatures (550–600 °C), this causes a significant decrease in the core hardness of standard MARAGING steels. This fact should be taken into account when the application of these processes is considered.

Plasma nitriding is the most suitable heat treatment process for the processing of MARAGING steels because the temperature of nitriding can be lower than (or equal to) the optimum temperature of ageing. Therefore, the process of ageing can be carried out during nitriding. As far as an improved resistance to abrasive, erosive and adhesive wear is concerned, the plasma nitriding of MARAGING steels proves to be more effective than solution annealing and aging, but still less effective than carburizing and boriding. It is to be expected that further improvements and the optimization of plasma nitriding parameters will produce layers with even a higher resistance to wear. In addition to the already mentioned advantages of the procedure, plasma nitriding results in some other favourable properties, such as an improved resistance to thermal fatigue, improved corrosion stability, and improved resistance to adhesion of the processed molten mixture. All this contributes to a conclusion that this procedure can be recommended in the manufacture of moulds made of MARAGING steels used for the processing of polymers and metals

Significant improvements in the resistance to erosion and adhesion of MARAGING steel 14 10 5 achieved by plasma nitriding followed by coating the metal by a TiN layer speak in favour of the application of this combination of processes for the manufacture of moulds that

have to meet the requirement of an increased wear resistance.

It is expected that laser beams used for the modification and coating of surfaces will find their application in the manufacture of tools and in the repair of worn tool surfaces made of MARAGING steels, particularly if compact and portable diode lasers are used.

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