

NANOMODIFICATION OF FORMING TOOL SURFACE BY ION IMPLANTATION

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ABSTRACT

In this paper the issue of ion implantation and its significance for ultra precision forming tools has been analyzed. An overview of ion implantation processes, influence of ion types and process parameters on enhancement of exploitation characteristics has been given. Some preliminary results have been given which have been conducted using high energy Kr ions obtained from an ECR (Electron Cyclotron Resonance) ion source. These ions have been used for nano modification of cold working steel. The obtained results clearly show that the energy of Kr ions has a great effect on the friction coefficient and roughness of the nano modified layer. An increase of nano hardness can be obtained as well. The preliminary results clearly show that ion implantation is a promising technology for nano modification of forming tools, however, in order to achieve the application of ion implantation throughout the industry a systematic approach to research is needed for the technology of ion implantation as well as for the connection between the exploitation characteristics and the type of applied ion implantation.

Keywords: *nanomodification, forming tool, ion implantation.*

1. INTRODUCTION

It has been recognized that many different tribological phenomena like wear (abrasive and/or adhesive), corrosion, friction, galling or sticking significantly reduce functionality and life time of forming tools. Thus, much effort has been put into solving such problems and it has become more and more obvious that many of these problems may be solved by surface modification treatments.

Among the best known are ion implantation, PVD (physical vapour deposition), PCVD (plasma chemical vapour deposition) and plasma nitriding. These techniques have very different characteristics, and they are capable of synthesizing many different surface treatments with specific tribological properties [1].

At the beginning of the eighties, ion implantation was believed to be a revolutionary surface treatment called to solve many wear and corrosion problems of metallic tools and components [2]. Their advantages with respect to other treatments allowed us to think that a relevant share of the market will be gained by the future ion implantation centers.

These advantages are certainly non-trivial. The stress has been put in the following facts:

1. Ion implantation is a low temperature treatment,
2. Ion implantation does not change neither dimensions nor surface finishing,
3. Ion implanted layers can not be delaminated: they are a part of the substrate itself,
4. Ion implantation can be focused to the areas to be protected, without, touching anything else. That allows efficient time of treatment and lower cost,
5. Ion implantation can be applied on surfaces previously treated by other techniques like PVD or CVD (chemical vapour deposition) deposition.

The growth of the ion implantation technology, has been slower than the predicted and much slower than the growth of other treatments like PVD coatings. The reasons for this situation could be explained with some characteristics of the ion implantation technology:

1. Ion implantation at the usual energies (lower than 200 keV) only affects a thickness less than 0,5 μm compared to 2-4 μm thickness of PVD coatings.
2. Directional treatments touch the samples one by one. The treatment time is proportional to the surface or to the number of components to be treated, unlike the PVD or CVD treatments that can treat big sets of samples simultaneously.
3. The line-of-sight character of ion implantation can make impossible the treatment of samples with complex shapes.
4. The time of treatment can not be reduced beyond some limits to prevent the excessive heating of the samples.

A specific problem for spreading the application of ion implantation throughout the industry was the fact that most companies knew nothing about the existence and advantages of ion implantation treatments. Deposition of PVD coatings results in visible changes of tool surfaces – in the sense of color, but changes made by ion implantation are usually not visible. A large campaign of spreading information was needed to make the companies aware of the new solutions provided by the advanced surface engineering. In this campaign many dissemination techniques were used: seminars, articles in industrial journals, videos, web pages and free trials to report case studies.

2. ULTRA PRECISION FORMING

Ultra precision forming is a new area which has been developing during the last few years. By analyzing the data from the SCOPUS (<http://www.scopus.com>) data base there can be found only 10 references concerning ultra precision forming, published in journals, but 86 registered and copyrighted patents can be found as well. This is evidence that this area is being greatly explored in the aim of getting technological advantage, however, the obtained results are relatively rarely published.

Why this happens? Ultra precision machining is gaining great importance throughout the globe and that is simply a result of the development trend of technology which had been set by Taniguchi fig. 1. According to fig. 1, nanometer precision has all ready been achieved a few years ago for ultra precision machining, however, precision machining and forming are expected to reach the nanometer scale precision during the next few years.

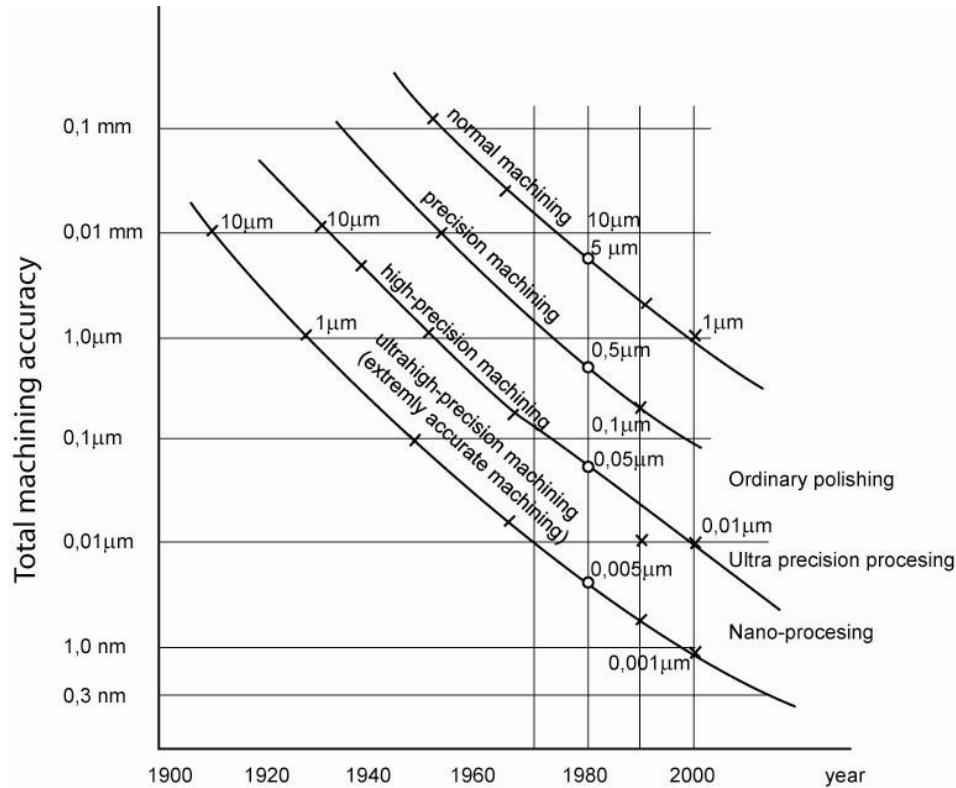


Figure 1. Trends in precision machining by Taniguchi [3]

Forming tool lifetime is directly dependant on surface phenomena which occur during exploitation, but most of all adhesive and abrasive wear. The usual trend of the tool dimension change is given in fig. 2, where it can be seen that in the first period of surface adjustment, roughness has a great influence on the wear phenomena. Therefore if nano precision forming is to be achieved, the tool surface must be modified in a way to lower roughness to nano scale values. The second wear period is characterized by the almost horizontal line of the tool dimension change which can be achieved only by improvement of mechanical properties of the surface layer. For the case of ultra precision forming it means that in the second period of wear, tool dimensions can be changed only by few tenths of a nanometer before the third wear period with a disastrous increase of wear rate starts. Therefore, in our further work we will be oriented at investigating those techniques which will improve roughness and mechanical characteristics of the tool surface layers with depths which do not exceed 100 nm.

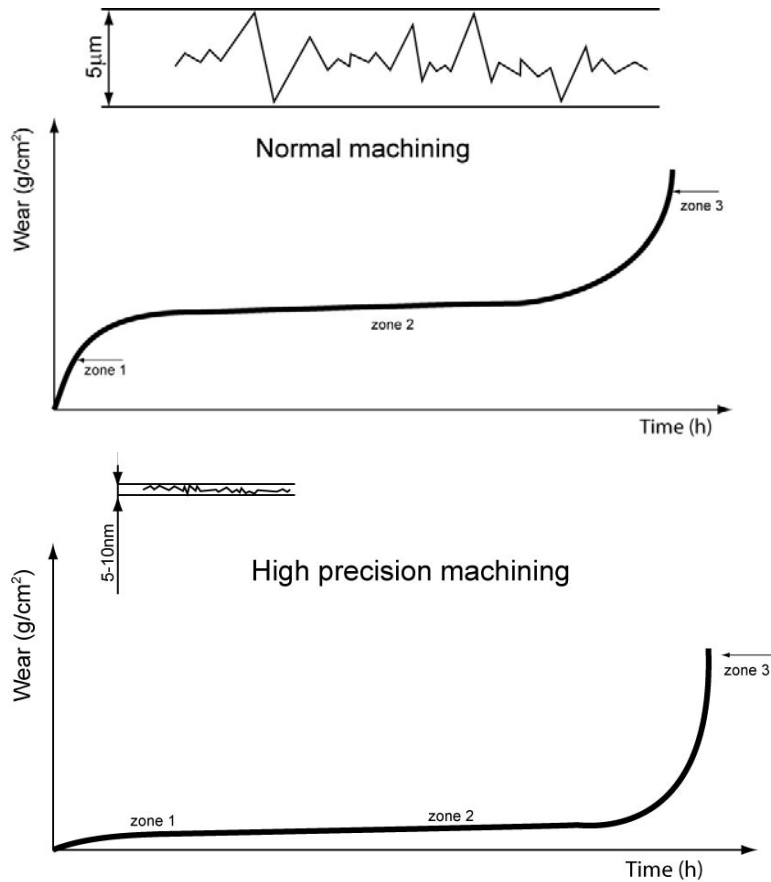


Figure 2. Normal machining, high precision machining

The electronic industry demands stamped parts with high performance. Therefore, punching tools like cutting punches with very high precision have to be used. To increase the lifetime of the punches made of steel, implantations with carbon, nitrogen, boron and titanium, and co-implantation with titanium and carbon were performed at energies from 50 keV to 200 keV and 600 keV and 700 keV with different doses in the region of several times 10^{18} cm^{-2} , measured perpendicular to the ion beam. A maximum increase in lifetime of a factor of 3,6 was reached. The surface roughness had a large influence on the increase in lifetime and the improvement caused by specific ion species [4].

As an example of precision forming in paper [5] dies for microgears were fabricated with FIB (focused ion beam), while die materials were glassy carbon (fig. 3(a)) and Zr-based metallic glass (fig. 3(b)). The gear module is 100 nm, the tooth thickness is 160 nm, and the addendum circle is 1 μm. Nanogears of Pt-based metallic glass have been die-forged with a die of glassy carbon (c) and with a die of Zr-based metallic glass (d). Traces of deformed and sheared gears are observed in fig. 3(c), although the phenomenon is not observed in fig. 3(d). Similar phenomena have been

observed in die-forging using glassy carbon dies and may be due to the difference in thermal expansion between the die and forged materials.

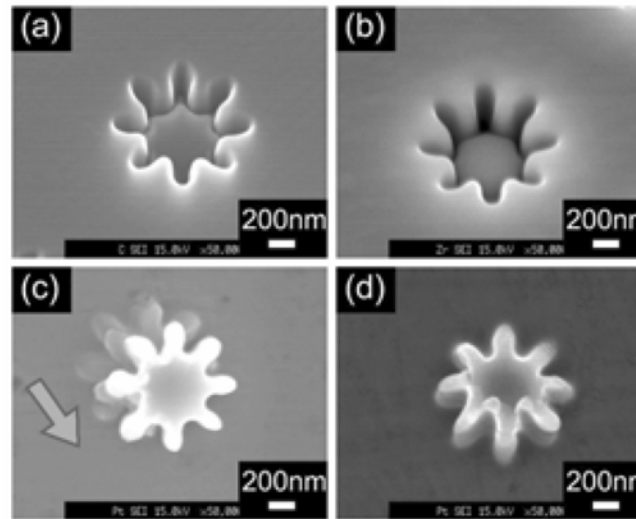


Figure 3. Die of (a) glassy carbon, (b) Zr-based metallic glass. Die-forged 1 micrometer diameter gear Pt-based metallic glass (c) with die (a) and (d) with die (b). Gear module=100 nm, $T_w=560$ K, $\sigma=20$ MPa, $t_w=400$ s

3. HOW TO CHOOSE THE TYPE OF THE ION IMPLANTATION PROCESS

Most often three types of solutions occur:

- Implantation of interstitial ions with small dimensions – N, C and B
- Implantation of metallic ions – Cr, Mo, Al, Ti and others.
- Implantation of ions of inert gases – Ar, Kr, Xe

Although the combinations of ions (single or double implantations), energies and doses makes the recipe book inexhaustible, the mutual experience for all treatment centers is that nitrogen implantation is almost the universal solution for ordinary problems, followed by carbon and chromium implantations.

Nitrogen implantation is actually the solution implemented in 80%-90% of all cases. Medium – high dose ($1\div 4 \times 10^{17}$ ions/cm²) nitrogen implantation of steels is widely employed for the most standard applications. Lifetime increases by three to five times.

Nitrogen ion implantation has generally been used to enhance hardness, wear resistance and frictional properties of steels [6]. Many of the benefits in wear resistance come from the increased surface hardness which is due to near-surface compound formation. In the case of nitrogen implantation, these compounds are nitrides and although the formation of iron nitrides may occur

in steels, it is usually the alloying additions that will form the hardest nitrides (e.g. aluminum, chromium, molybdenum, vanadium, titanium and tungsten).

Gas (primarily nitrogen) and metal ions are implanted to doses in the range 10^{16} – 10^{18} ion/cm². Nitrogen is currently used for two reasons. Firstly, it is relatively simple to obtain a gas ion beam where the beam parameters can be varied over a wide range. Secondly, most tools and component parts are made from steel containing nitride-forming elements. In contrast, metal ion implantation (MII) is not used so widely.

It is worth mentioning possible applications of low dose implantation. If doses below 10^{16} ions/cm² were effective enough to produce the desired changes, these solutions would be between 20 and 100 times cheaper than the ordinary II treatments. Rare earth metal ion implantation is very effective for protecting stainless steel and other metals against oxidation at high temperatures.

In fig. 4 the influence of the ion implantation process on wear resistance is shown.

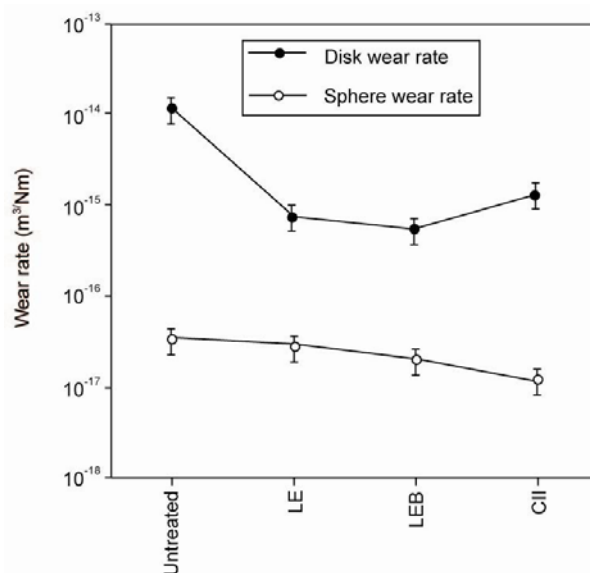


Figure 4. O1 tool steel discs and counter face steel sphere sliding wear rates for various treatments: LE-low energy implantation; LEB-low energy implantation with nitrogen backfill; CII-conventional ion implantation

There are a number of advantages which might be gained if low energy ion implantation treatments could be shown to be effective in modifying the surface mechanical properties of materials. Higher beam currents could then be used and faster treatments could be possible.

3.1 Metal ion implantation

Recently, a series of metal ion sources based on vacuum metal-arc have been developed to treat tools and components. These metal ion sources enable rapid treatment while maintaining good

surface quality of parts [7]. Some examples of the applications of metal ion implantation for increasing lifetime of tools and components are given in tab. 1.

The change in the surface properties after an ion-beam treatment results in changes in the microstructure and phases present in the surface of the implanted material. The effect of the ion beam is not limited to the alloyed surface layer (the implanted zone, IZ) but extends to a greater depth. A dense dislocation network is developed, termed the implantation affected zone (IAZ), which hardens the part and enhances the wear resistance.

Table 1. Some examples of the applications of metal ion implantation for increasing lifetime of tools and components.

Tool or component	Application	Lifetime Increase	Cathode
Drills, Ø 2,2mm	Cast iron	2,5 times	LaB6
Cam-shafts, valves, pistons, piston rings	various	2÷3 times	Mo
Punching dies	10KP steel	3 times	Mo
Punching dies	Stainless steel	4 times	Mo
Cutting dies	Aluminum foil	31 times	TiB2
Guillotine blades	Rubber	10÷16 times	TiB2
Cemented carbide inserts	Stainless steel	3,2 times	C

Applications of metal ion implantation include:

- a) the possibility of introducing any element into the surface of the substrate and forming alloys which are not predicted by the phase diagram,
- b) independence from any processes occurring in the bulk material,
- c) no adhesion issues between the implanted layer and the bulk,
- d) the process is carried out at relatively low temperatures,
- e) the geometrical dimensions and surface finish of the processed component remain unchanged,
- f) the depth distribution of the implanted element can be controlled,
- g) high productivity and reproducibility of the process.

3.2 SRIM simulation

Using the SRIM (Stopping and Range of Ions in Matter) simulation it can be calculated that the depth of surface implantation for 100Cr6 steel reaches about 70 nm for Kr^{8+} and about 90 nm for Kr^{11+} . Fig. 5 shows the result of the SRIM simulation of Kr^{8+} ions into 100Cr6 steel while fig. 6 shows the ion range distribution from the same experiment.

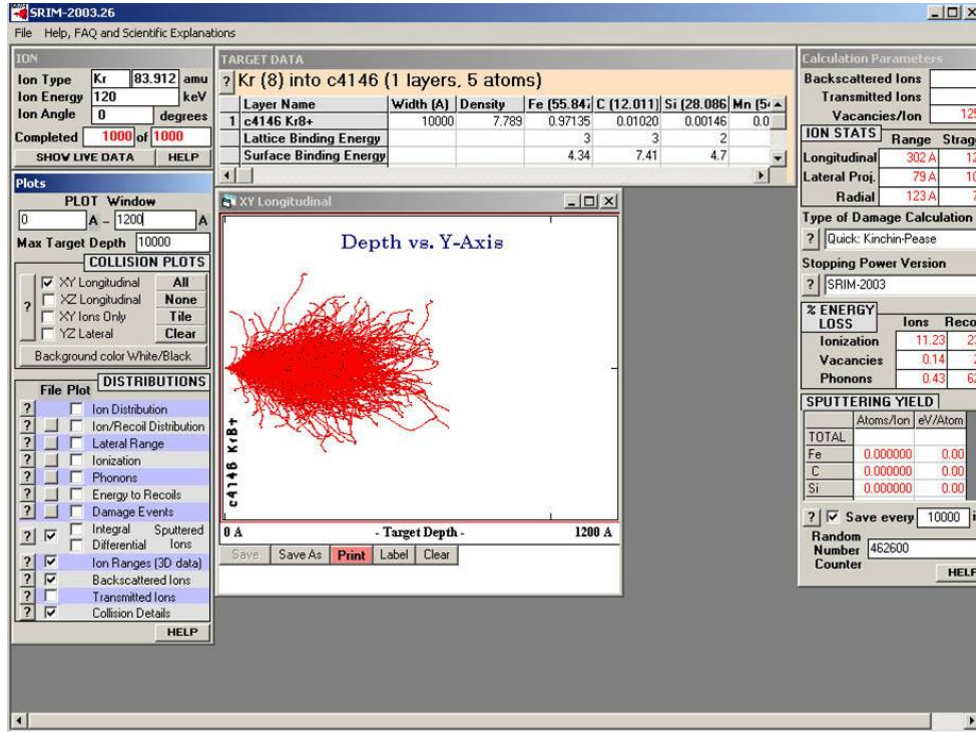


Figure 5. SRIM simulation of Kr^{8+} ions into 100Cr6 steel

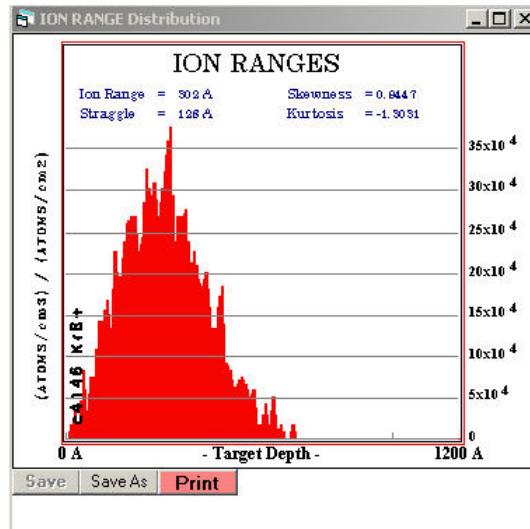


Figure 6. Ion range distribution

4. EXPERIMENTAL PROCEDURE

In Vinča there is a team of scientists who have managed to evaporate and ionize Kr. Those ions are mainly used for modification of materials used for semiconductors and superconductors. There are only a few laboratories in the world who possess equipment for Kr implantation and we are familiar with only a few attempts of Kr implantation into steel - and that has been done for medical implants made of stainless steel. Therefore, we have been eager to test, for the first time, the possibility of Kr ion implantation into cold working tool steel.

Krypton ions have been implanted in steel substrates using mVINIS Ion Source [8]. The mVINIS Ion Source shown in fig. 7, is a part of the TESLA Accelerator Installation (AIT) at the VINČA Institute of Nuclear Sciences in Belgrade.

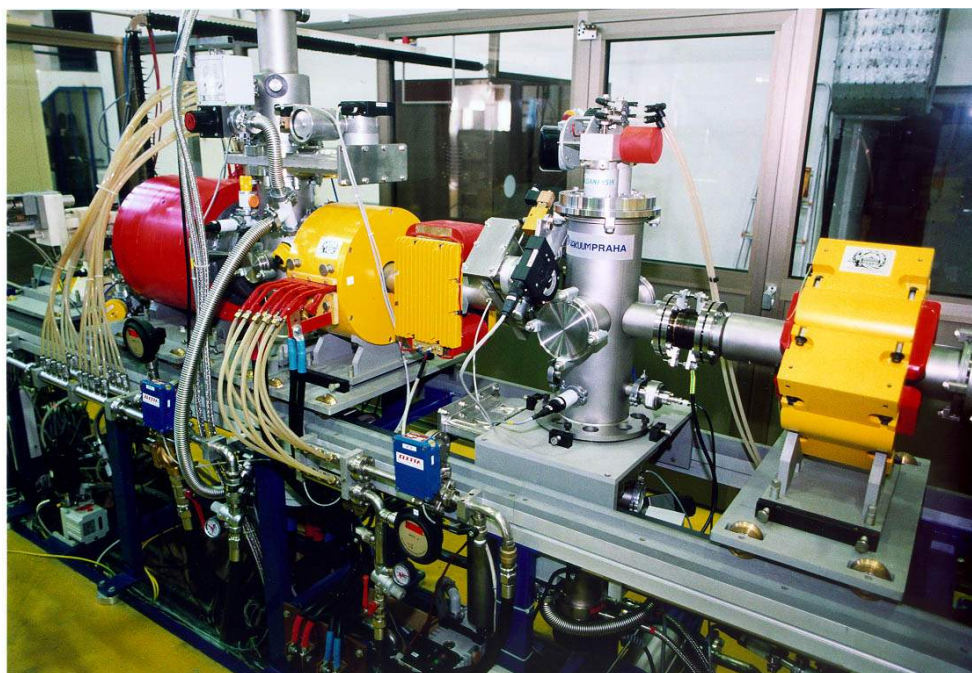


Figure 7. mVINIS Ion Source

Basic characteristics of the mVINIS ECR ion source are: operating frequency: 14,5 GHz, total power consumption of ECR ion source: 68 kW, radial plasma confinement: permanent hexapole magnet NdFeB, $B_r=1,24$ T, axial plasma confinement: electromagnet with two coils, max current 1000 A, mirror ratio: $B_{max}/B_{min}=1,29$ T/0,46 T=2,8, gas inlet system: fine flow control of main gas and supporting gas, solid substance inlet system: microoven inserted into the plasma chamber, $T_{max}=900^{\circ}\text{C}$, extraction system: simple two electrode system, plasma chamber at high voltage, $U_{ex}=5\pm 25$ kV, bias electrode: fixed position inside plasma chamber, $U_{bias}=0\pm 500$ V.

The mVINIS Ion Source can produce multiply charged ions from gases using a specially designed gas inlet system. This system is crucial for the stable and reproducible operation of the complete ion source.

In this paper Kr^{8+} ions with the energy of 120 keV and Kr^{11+} ions with the energy of 180 keV were used. The Krypton spectrum is shown in fig. 8.

Main parameters of ion implantation:

Parameters for Kr^{8+} - $U_{\text{ex}}=15$ kV; $I_{\text{ex}}=3,2$ μm ; $D=10^{16}$ ions/ cm^2 ; $W=120$ keV; $t=295$ min

Parameters for Kr^{11+} - $U_{\text{ex}}=16,4$ kV; $I_{\text{ex}}=4,14$ μm ; $D=10^{16}$ ions/ cm^2 ; $W=180$ keV; $t=280$ min

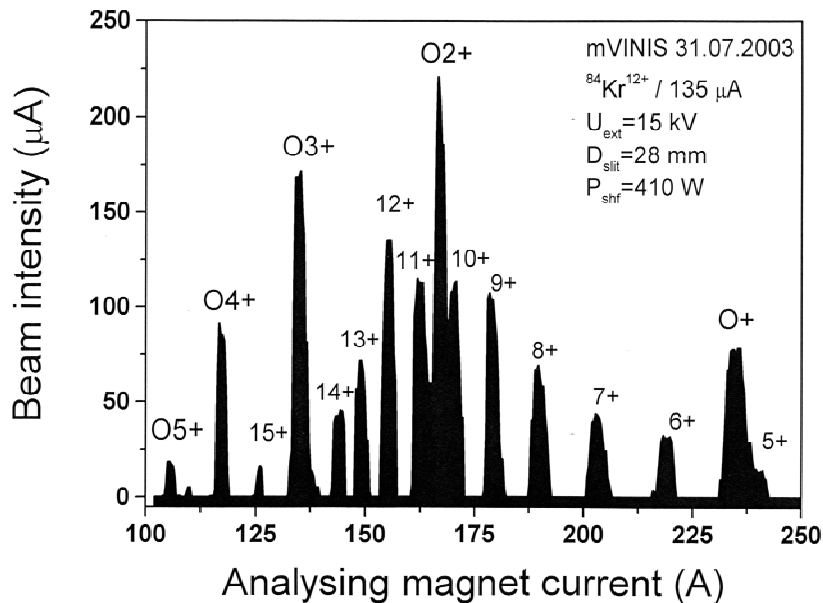


Figure 8. Krypton spectrum

5. RESULTS AND DISCUSSION

Surface roughness was measured by AFM SOLVER-P47 at BIONT a.s., Department of Nanotechnologies, Bratislava (fig. 9). Before implantation sample surfaces were grinded and polished.

Friction coefficient was measured with different load and type of prism – hard metal and diamond. Fig. 10 shows the influence of implanted ion energy on the friction coefficient for cold working tool steel 100Cr6. It could be concluded that ion implantation can decrease friction coefficient for both energies of Kr ions [9].

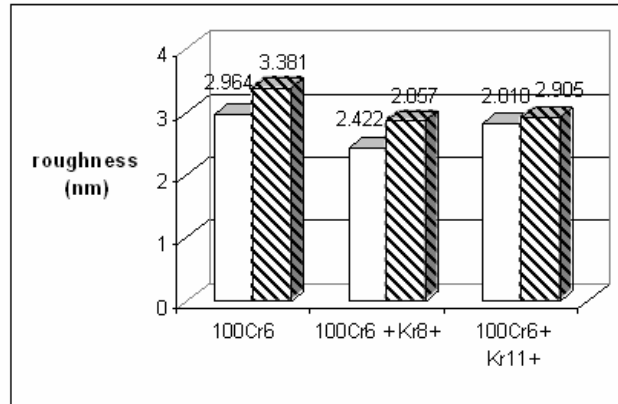


Figure 9. AFM surface roughness – 100Cr 6 steel

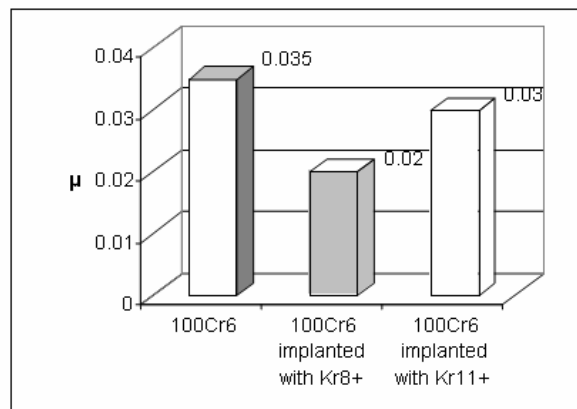


Figure 10. Friction coefficient μ - steel 100Cr 6, $F=10\text{ N}$

ERDA (Elastic Recoil Detection Analysis) analyses were provided by Dr. Wolfgang Bohne at Hahn-Meitner Institut, Berlin [10]. All samples were measured with a beam of Au ions (26^+) with an energy of 350 MeV and a beam intensity of about 80 particle pA. The detection angle was 58° .

Results of ERDA show that all samples have an oxide layer with some H and C at the surface, assumed as Fe_2O_3 or similar – fig. 11 and fig. 12. The weak steel components close to Fe like Mn, Cr and V were not separately evaluated. They are included in the concentration of Fe. All concentrations are given as atom%. Thicknesses are areal densities of ions in at/cm^2 . They can be converted into geometrical depth if the density of the material is known.

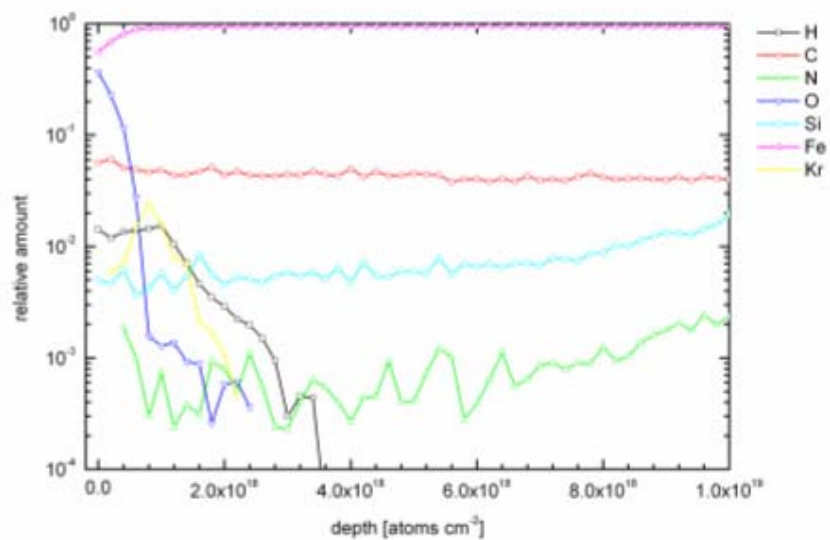


Figure 11. Results ERDA analyses for sample – Kr^{11+}

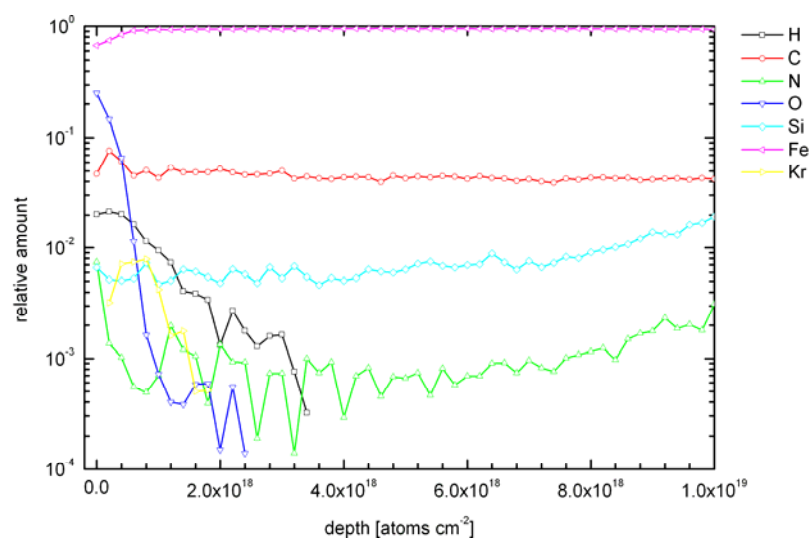


Figure 12. Results ERDA analyses for sample – Kr^{8+}

The indentation tests were carried out in the low load range of the NanoTest Platform in laboratory L.O.T.-Oriol GmbH & Co Darmstadt, Germany. A three-faced Berkovich diamond indenter (which has the same projected area-to-depth relation as a Vickers indenter) was used. All

the tests were carried out using a depth control mode, i.e. the loading process was finished and reversed at a pre-set peak depth. Fig. 13 shows results of hardness measurements for Kr^{11+} implanted 100 Cr 6 steel.

The loading rate was chosen so that the maximum load was reached after 20 seconds loading. A dwell time (i.e. peak load holding period) of 10 seconds was applied to all the tests. Due to statistical reasons, we did seven indents with the same load. Over these seven indents we calculated the mean value for the result plots.

Mean value and the standard deviation have been calculated for seven individual experiments tab. 2 and tab. 3.

Table 2. Statistical results on sample Kr^{11+} 100 Cr 6 un-implanted

Max. Load	Plastic Depth	Hardness		Er		Plastic Work	Elastic Work
(mN)	(nm)	(GPa)	std. dev.	(GPa)	std. dev.	(nJ)	(nJ)
1	31,9	53,90	10,45	427,23	32,06	0,012	0,010
2	55,2	32,06	5,34	347,65	21,29	0,042	0,026
4	96,4	19,58	2,72	309,33	15,07	0,138	0,070
6	132,5	15,21	2,80	289,00	13,84	0,288	0,122
8	157,1	14,32	4,98	246,31	33,10	0,448	0,197
10	185,5	12,74	0,65	196,06	8,56	0,618	0,312
15	236,8	11,62	1,88	186,93	2,01	1,176	0,576
20	287,6	10,45	0,87	177,30	8,61	1,816	0,872
25	309,0	11,31	0,68	174,60	4,83	2,283	1,273

Table 3. Statistical results on sample Kr^{11+} 100 Cr 6 steel implanted

Max. Load	Plastic Depth	Hardness		Er		Plastic Work	Elastic Work
(mN)	(nm)	(GPa)	std. dev.	(GPa)	std. dev.	(nJ)	(nJ)
1	26,9	81,940	5,616	497,815	39,526	0,009	0,011
2	57,7	28,939	1,770	336,503	9,117	0,043	0,027
4	89,2	23,065	2,722	285,967	20,294	0,121	0,077
6	118,5	19,296	1,801	264,593	20,040	0,245	0,141
8	138,7	19,195	1,071	282,289	6,918	0,374	0,207
10	175,0	14,303	0,720	248,763	9,348	0,606	0,274
15	223,5	13,175	2,077	241,388	22,315	1,095	0,479
20	267,8	12,111	8,248	236,707	64,164	1,718	0,738
25	292,5	12,644	0,733	245,499	10,293	2,173	1,032

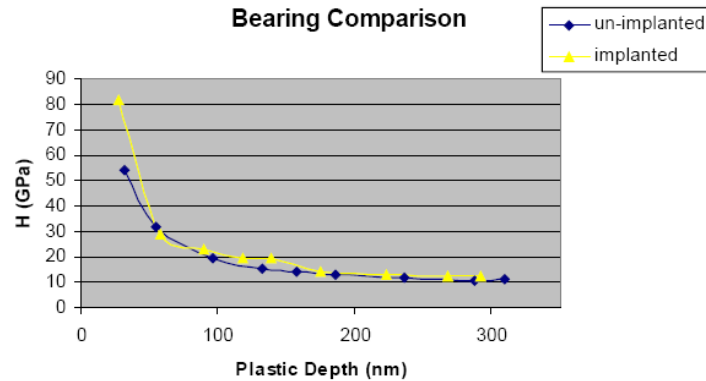


Figure 13. Kr^{11+} implanted 100 Cr 6 steel - comparison regarding hardness

Based on these results it can be concluded that the surface layer with nano dimensions has an effect on nanohardness increase due to ion implantation.

6. PLAN FOR FURTHER INVESTIGATION

Contact friction represents resistance to relative movement of two bodies in contact where normal stress acts between them. Since there is always relative movement between the die and the material in plastic deformation process, friction is an integral part of every deformation processes (exception is uniaxial tension). Contact friction is a negative event because it arises increase of deformation force and work, die wear and uneven deformation.

For quantitative manifestation of friction value, friction coefficient (μ) and friction factor (m) are utilized. These values are used during definition of tangential stress that appears because of the friction on contact surfaces. In spite of that, there are three models of tangential stress (τ_k) expression that arises because of the friction: a) $\tau_k = \mu \cdot \sigma_n$ b) $\tau_k = \mu \cdot K$ and

c) $\tau_k = m \cdot \tau_{\max}$, where σ_n is normal stress and K is flow stress. Relation between friction factor and friction coefficient is expressed with equation $m = \mu\sqrt{3}$ [11].

Magnitude of friction coefficient (μ), i.e. friction factor (m) is possible to determine experimentally by various methods [12]. One of the most applicable methods is ring (hollow cylinder with low height) upsetting.

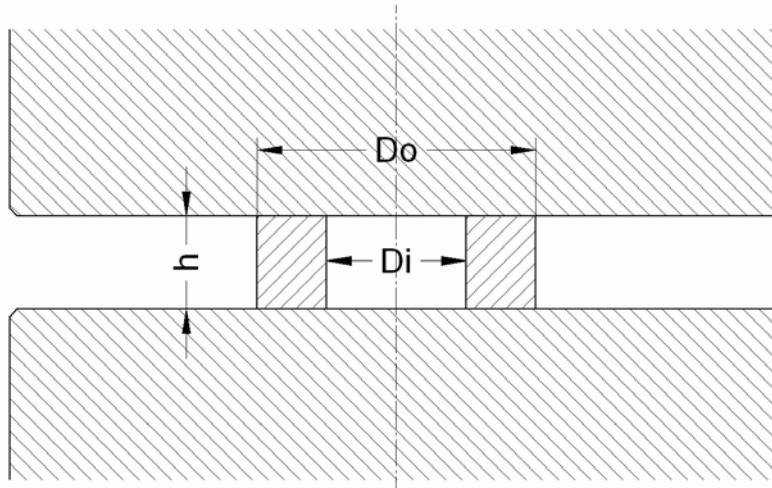


Figure 14. Ring upsetting process scheme

For researching the influence of implantation on contact friction, analysis of friction coefficient is planned on dies made of steel Č. 4150 (DIN: X 210 Cr 12) implanted with:

- Yr, Hf, Al and N
- various ion energies
- different types of implantation – high energy ions and low energy, high flux ions
- different initial roughness
- different base material – steel and steel with a TiN coating

Experiments of ring upsetting by flat plates will be conducted on specimens with initial dimensions $D_o=18$ mm, $D_i=9$ mm and $h=6$ mm made of steel Č. 1221 (DIN: Ck 15) according to process scheme on fig. 14.

7. CONCLUSION

Ion implantation is a very important technique for surface nano modification especially for forming tools. With high consideration in choosing the type of ion implantation, types of ions and main parameters (temperature, energy, incident angle ect.), planned modifications of the surface layers can be achieved, having in mind that the results greatly depend on the base material as well.

Kr ions with desired energies, but with relatively low fluxes, which could present a problem for a wider industrial application, have been obtained in the ECR ion source. This is in the sense of longer duration of the process of ion implantation which rises the price of the modification of the forming tool surface.

After our preliminary results gained from the testing of the Kr ion implanted surfaces of a cold working steel it can be concluded:

- a) Ion implantation of Kr could decrease surface nanoroughness for cold working steel 100Cr 6, but results depend on the Kr ion energy.
- b) ERDA profile shows significant differences in nano surface zone composition, depending on the applied type of implanted Kr ion.
- c) Friction coefficient measured with $F=10\text{N}$ shows some correlation to surface roughness for cold working steel 100Cr 6.

In order to obtain more actual results about the applicability of ion implantation for nano modifications it is necessary to make a very detailed, carefully planned experiment with variations of the most important process parameters, ion types and types of substrate surfaces (initial roughness, hardness, composition etc.) in order to successfully solve the problem of ultra precision forming tool production.

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NANOMODIFIKACIJE POVRŠINA ALATA ZA DEFORMISANJE JONSKOM IMPLANTACIJOM

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REZIME

U ovom radu analizirana je jonska implantacija i njen značaj za alate za ultra precizno deformisanje. Dat je pregled procesa jonske implantacije kao i uticaj vrste jona i parametara procesa na povećanje eksploatacionih karakteristika. U radu su prikazani i neki preliminarni rezultati koji su dobijeni korišćenjem jona Kr visoke energije iz ECR jonskog izvora. Ti joni su iskorišćeni za nanomodifikacije čelika za rad u hladnom stanju. Dobijeni rezultati jasno pokazuju da energija jona Kr ima veliki uticaj na koeficijent trenja i hrapavost nanomodifikovane prevlake. Takođe se može dobiti i povećanje nano tvrdoće. Preliminarni rezultati jasno pokazuju da je jonska implantacija značajna tehnologija za nano modifikacije alata za deformisanje. Međutim, da bi se postigla primena jonske implantacije u industriji, potreban je sistematski pristup u istraživanju tehnologije jonske implantacije kao i istraživanje veze između eksploatacionih karakteristika i vrste jonske implantacije.

Kako se pri obradi plastičnim deformisanjem uvek pojavljuje relativno kretanje između alata i materijala, trenje je sastavni deo svakog procesa deformisanja. Kontaktno trenje je negativna pojava, jer utiče na povećanje potrebne sile i rada deformisanja, habanje alata kao i na neravnomernost deformacije. Radi toga je neophodno odrediti koeficijent kontaktnog trenja μ , odnosno faktor smicanja m .

Jedna od najčešće primenjivanih metoda za određivanje veličine koeficijenta kontaktnog trenja jeste metoda sabijanja prstena koja će biti korišćena za potrebe ispitivanja uticaja jonske implantacije na kontaktno trenje. Planirana su ispitivanja koeficijenta trenja na alatima implantiranim Yr, Hf, Al i N, različitim energijama jona, različitim tipovima implantacije, na površinu različite početne hrapavosti i na osnovni materijal od čelika i čelika sa TiN prevlakom.

Ključne reči: nanomodifikacija, alati za deformisanje, jonska implantacija.