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NANOMODIFICATION OF TOOL STEEL BY ION IMPLANTATION

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ABSTRACT
The importance of nanomodifications in technology development has been growing, especially in development of new generation of tools. The development trend of precision and ultra precision machining clearly shows that we are dealing with up to few tens of nanometers in size. Therefore, depth of modification of tools should be between 30-100nm. In this paper, we will analyze the possibility of nanomodification with the aim of obtaining high quality surface layers on tool steels. In addition, we will give some preliminary results of investigations of possibilities of nanomodification by Kr ion implantation.

KEYWORDS
Nanomodification, ion implantation, Kr, ultra precision machining

1. INTRODUCTIONS

Ultra precision machining is a new area which has been developing during the last decade. Around 460 papers published in journals and about 2260 registered and copyrighted patents concerning ultra precision machining can be found by analyzing available databases. The number of patents compared to the number of papers testifies that this area is being greatly explored with the aim of getting technological advantage. It should be pointed out that 358 papers are related to engineering, 105 are related to material science and only 77 are related to physics.

Ultra precision machining has been getting great importance throughout the globe. This is simply the result of the technology development trend set by Taniguchi [1]. According to this reference, nanometer accuracy has already been achieved in ultra precision machining. However, precision machining and forming are expected to reach the nanometer scale accuracy during the next few years.

Tool lifetime depends directly on the surface phenomena which occur during exploitation, mainly on adhesive and abrasive wear. The usual trend of the tool dimension change is given in Figure 2. 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 machining 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 in the change of tool dimension, which can be achieved only by improvement of mechanical properties of the surface layer. In the case of ultra precision machining, 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 decrease roughness and improve mechanical characteristics of tool surface layers with depths bellow 100 nm.

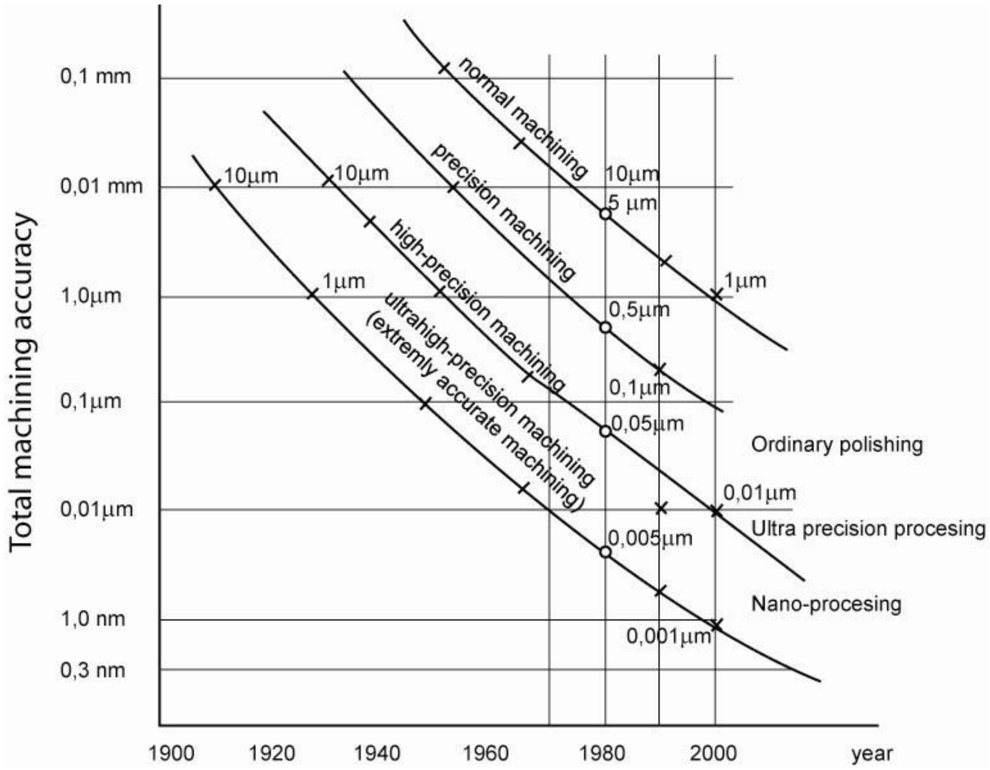

Figure 1. Trends in precision machining by Taniguchi [1]

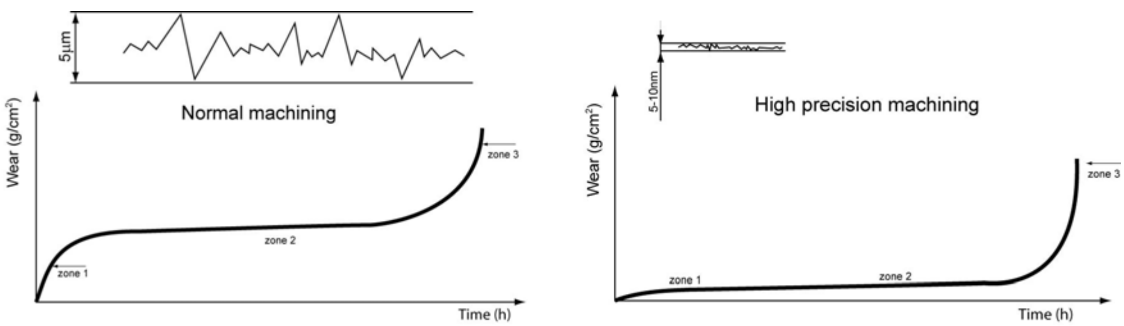

Figure 2. Tools wear during normal and high precision machining

Where do we need such precise tools? For example: the electronic industry demands stamped parts with high performances. Therefore, punching tools like cutting punches with very high precision have to be used. The lifetime of the punches made of steel can be increased by implantation and co-implantation of various types of ions at various energies and doses. Carbon, nitrogen, boron and titanium ions were implanted and co-implanted at energies between 50 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 improvement of surface roughness had a large influence on the increase in tool lifetime [2].

It has been recognized that many different tribological phenomena like wear (abrasive and/or adhesive), corrosion, friction, galling or sticking significantly reduce functionality and lifetime of 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 techniques are ion implantation, PVD (physical vapor deposition), PCVD (plasma chemical vapor deposition) and plasma nitriding. These techniques have very different characteristics, and they are capable of synthesizing many different surface treatments with specific tribological properties [3].

At the beginning of the eighties in the 20th century, ion implantation was believed to be a revolutionary surface treatment called to solve many wear and corrosion problems of metallic tools and components [4]. Its 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 centres. These advantages are certainly non-trivial. Ion implantation is a low temperature treatment which does not change dimensions. Ion implanted layers can not be delaminated because they are a part of the substrate itself. Significant advantage is the ability to focus to the areas which are to be protected, without affecting other areas. That allows efficient time of treatment and lower costs. Also, a particular advantage is that ion implantation can be applied on surfaces previously treated by other techniques like PVD or CVD (chemical vapor deposition).

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

1. Ion implantation at the usual energies lower than 200 keV affects only a thickness of material less than 0.5 μm compared to 2÷4 μm thickness of PVD coatings.
2. Directional treatments process 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, ion implantation treats only one sample at the time.
3. The line-of-sight character of ion implantation can make the treatment of samples with complex shapes impossible.
4. The time of treatment can not be reduced beyond some limits in order 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 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.

If it is taken into account that ultra precision tools require nano modifications in the range of 100nm, ion implantation appears to be not only the most appropriate technique, but the only possible solution having in mind the type of machining, type of tools, as well as type of exploitation conditions.

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 ($1\div4x10^{17}$ ions/cm^2) 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 [5]. Many of the benefits in wear resistance come from 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 $10^{16}\div10^{18}$ ion/cm^2. Nitrogen is currently used for two reasons. First, it is relatively simple to obtain a gas ion beam where the beam parameters can be varied over a wide range. Second, 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/cm^2 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.

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.

2. METHODOLOGY

Krypton ions have been implanted in steel substrates using mVINIS Ion Source. The mVINIS Ion Source shown in figure 3 is a part of the TESLA Accelerator Installation (AIT) whose construction is in the final stage at the VINCA Institute of Nuclear Sciences in Belgrade. The major parts of this facility are an isochronous cyclotron (VINCY), a heavy ion source (mVINIS), a light ion source (pVINIS) and a number of experimental channels. The mVINIS Ion Source is an ECR (electron cyclotron resonance) ion source (figure 4) with multiple applications. It can serve as an injector of the VINCY Cyclotron providing heavy ions for several high-energy experimental channels (in the field of radiation physics, radiation biology, physics of thin crystals, nuclear physics). On the other hand, it can also work as a stand alone machine, directly delivering ion beams to the low energy experimental channels (physics of multiply charged ions, surface physics, and modification of materials by ion beams).

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: micro oven inserted into the plasma chamber, T_{max} = 900 °C, extraction system: simple two electrode system, plasma chamber at high voltage, U_{ex}=5 ÷ 25 kV, bias electrode: fixed position inside plasma chamber, U_{bias}= 0 ÷ - 500 V.

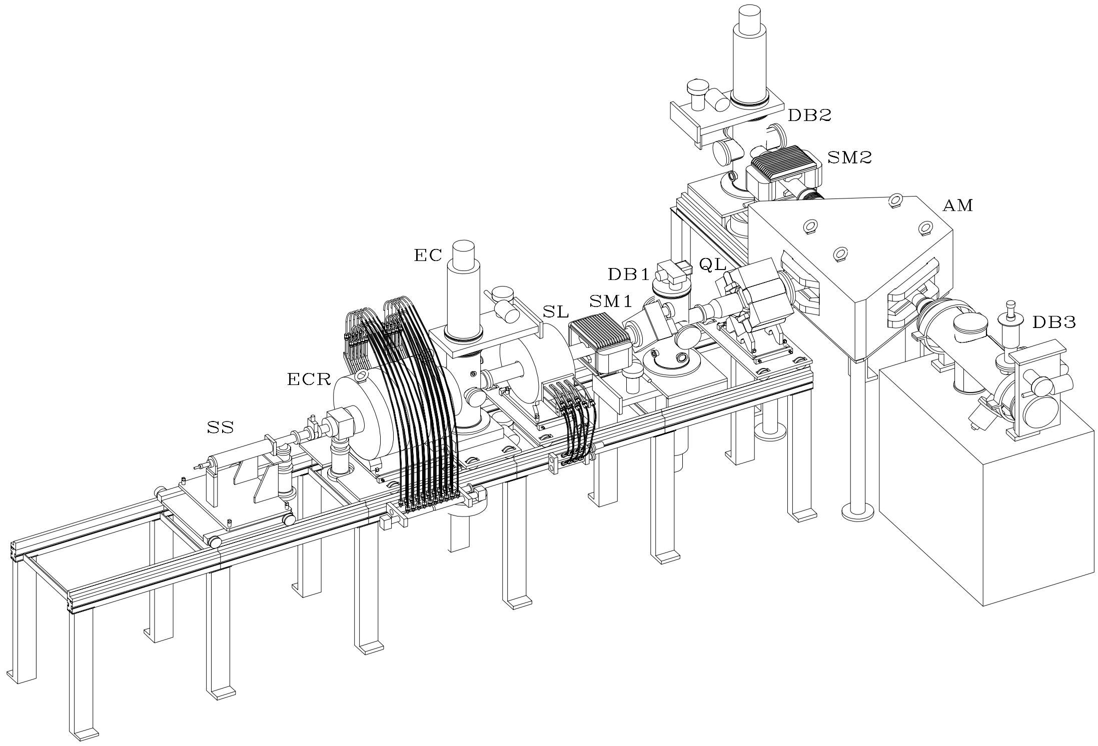

Figure 3. mVINIS Ion Source as a complete heavy ion injector.
(ECR – ECR ion source; SS – solid substance inlet system; EC – extraction chamber; DB1, DB2, DB3 – diagnostic boxes; SL – solenoid lens; SM1, SM2 – steering magnets; QL – quadrupole lens, AM – analyzing magnet).

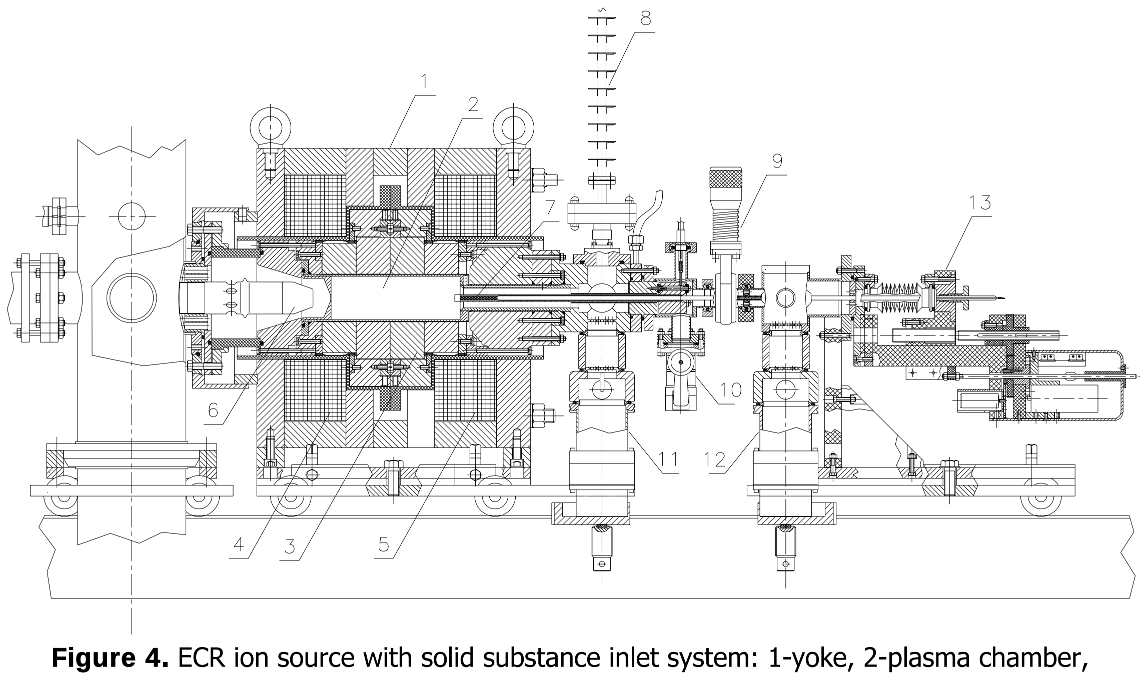

Figure 4. ECR ion source with solid substance inlet system: 1-yoke, 2-plasma chamber, 3-hexapole, 4-extraction stage coil, 5-injection stage coil, 6-extraction electrode, 7-minioven installed inside the bias electrode, 8-waveguide, 9-gate valve, 10-gas dosing valve, 11 and 12-turbo- molecular pumps, 13-minioven positioning system

It consists of an ECR ion source working at 14.5 GHz, gas and solid substance inlet systems, focusing and analyzing magnets along the beam transport line, vacuum system, ion beam diagnostic system, safety and control systems and utilities (water cooling system, compressed air system etc.). The major parts of the ECR ion source with the installed solid substance inlet system are shown in Figure 4.

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. The gas inlet system introduces the main gas and, if required, the additional (supporting) gas into the plasma chamber. It is based on two separate gas dosing lines enabling the fine adjusting of very low gas flows, in the range 0.1-10 cm^3/h. The automatic gas dosing valves (Balzers UDV140/RVG050B) keep the gas flow constant irrespective of the temperature and pressure variations in the gas containers. The vacuum gauge measuring the pressure at the entrance of the injection chamber produces the pressure-dependent electrical signal and transmits it to the RVG050B control unit. The control unit (PI controller) regulates the gas flow through the UDV140 gas dosing valve to ensure that the desired pressure in the vacuum chamber is maintained.

In our work we had used two different types of steel. The reason was that these steels have different machinability due to the difference in steel structure caused by the different percentage of carbon and alloying elements present. Since the structure is not the same, the effects of the implantation will also not be the same. The base of the 100Cr6 steel is consisted of uniform martensite so we decided to present only these results in this paper. The stopping of ions in a material as uniform as steel 100Cr6 will be on a different depth comparing to the material with a non-uniform structure like for example high speed steel - M2.

In this paper Kr^{8+} ions with the energy of 120 keV and Kr^{11+} ions with the energy of 180keV were used. The Krypton spectrum is shown in figure 5.

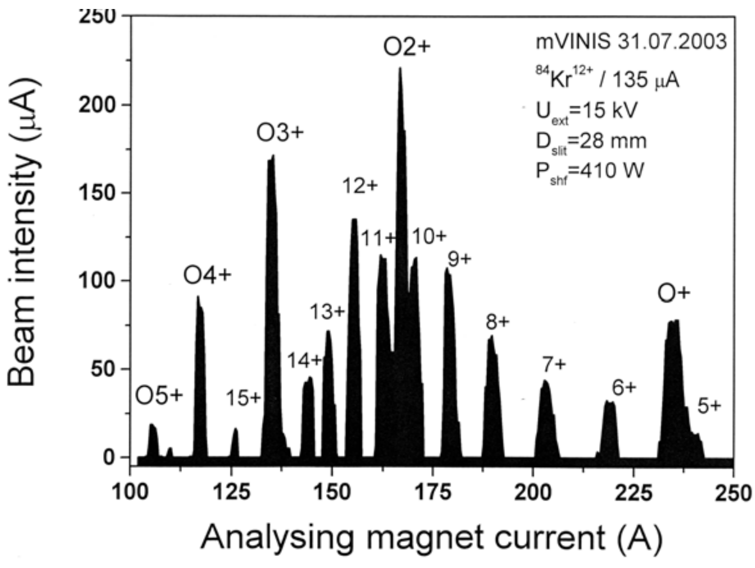

Figure 5. Krypton spectrum

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+}. Figure 6 shows the result of the SRIM simulation of Kr^{8+} ions into 100Cr6 steel while figure 7 shows the ion range distribution from the same experiment.

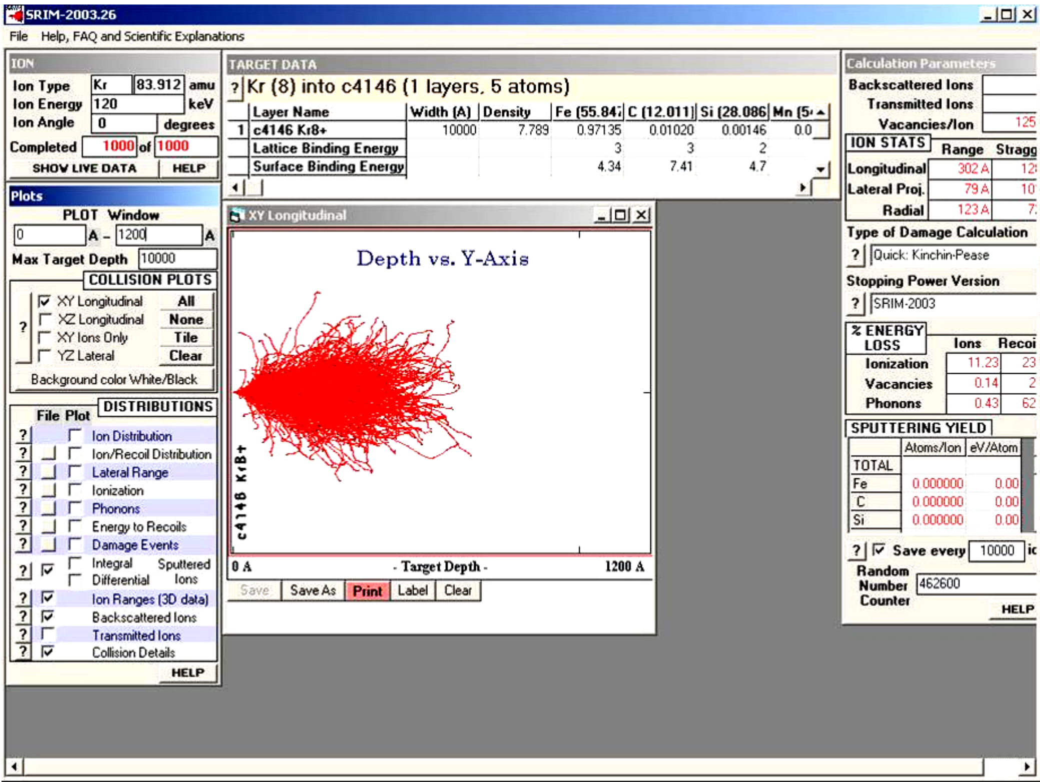

Figure 6. SRIM simulation of Kr^{8+}ions into 100Cr6 steel

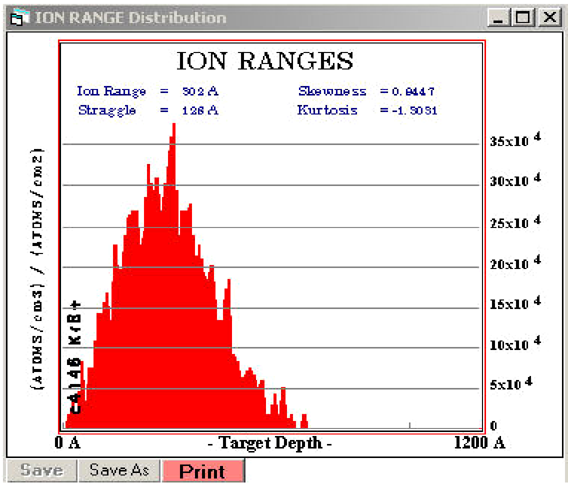

Figure 7. Ion range distribution

3. RESULTS AND DISCUSSION

The results of friction coefficient measurements are presented in the table 1. Two different loads were applied and significant differences in friction coefficients were obtained. Results presented in the table 1 are average values of several measurements. Hard metal was used as a counter material during measurements.

Table 1. Friction coefficients

Steel	Friction coefficient μ, F=10N	Friction coefficient μ, F=100N
100Cr6	0.035	0.09
100Cr6 implanted with Kr^{8+}	0.02	0.062
100Cr6 implanted with Kr^{11+}	0.03	0.075

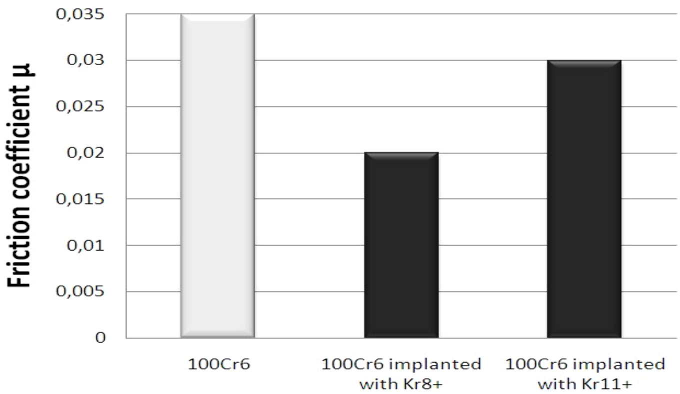

Figure 8. Friction coefficients for F=10N

It is obvious from the figure 8 that friction coefficient decreased for all applied energies of Kr ions. This phenomenon is in accordance with the literature referred in the introduction.

Change in the friction coefficient can be explained with the decreased surface roughness, as shown in the figure 9. Typical surface image measured by AFM SOLVER-P47 is presented on figure 10.

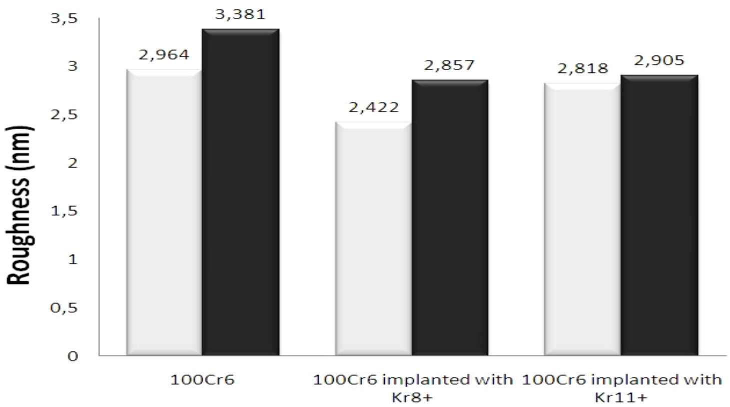

Figure 9. Surface roughness measurements for 100Cr6 steel (minimum and maximum measured values)

The indentation tests were carried out in the low load range of the NanoTest Platform in laboratory L.O.T.-Oriel 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. Figure 11 shows results of hardness measurements for Kr^{11+} implanted 100 Cr 6 steel. The loading rate was chosen in the way that the maximum load was reached after 20 seconds. A dwell time (i.e. peak load holding period) of 10 seconds was applied in all tests. Due to statistical reasons, seven indents with the same load were made. Over these seven indents we calculated the mean value for the result plots.

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.

ERDA (Elastic Recoil Detection Analysis) analyses were provided by Dr. Wolfgang Bohne at Hahn-Meitner Institut, Berlin [6]. 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 obvious difference between distributions of Kr at sample surface bombarded with different Kr ion energies (different Kr ions).

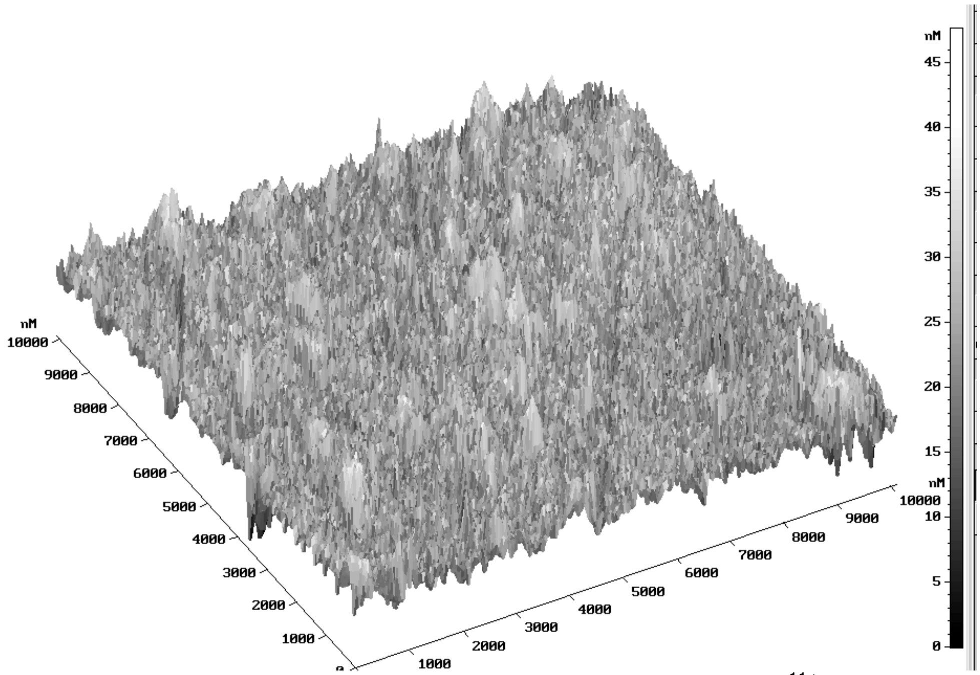

Figure 10. AFM image 100Cr6 steel implanted with Kr^{11+}

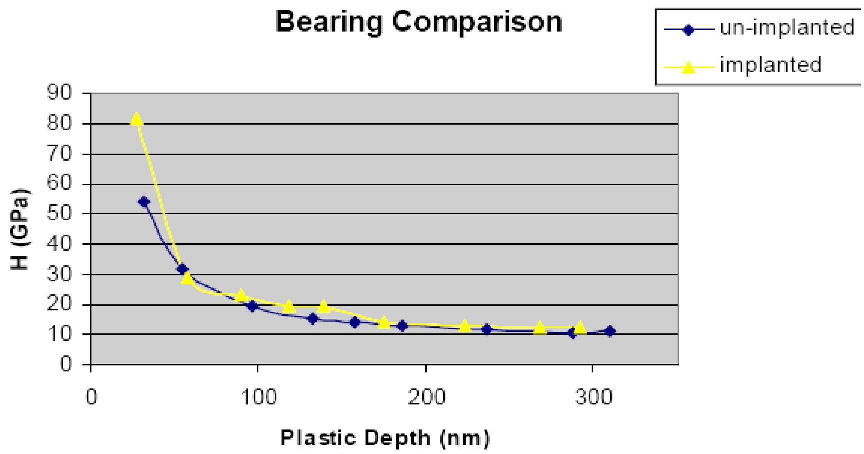

Figure 11. Kr^{11+} implanted 100 Cr 6 steel - comparison regarding hardness

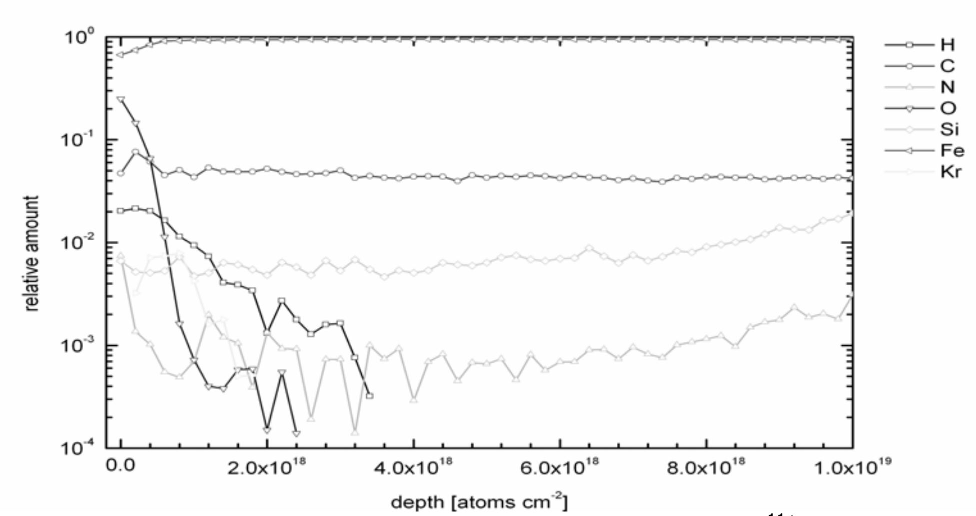

Figure 12. Results ERDA analysis for sample – Kr^{11+}

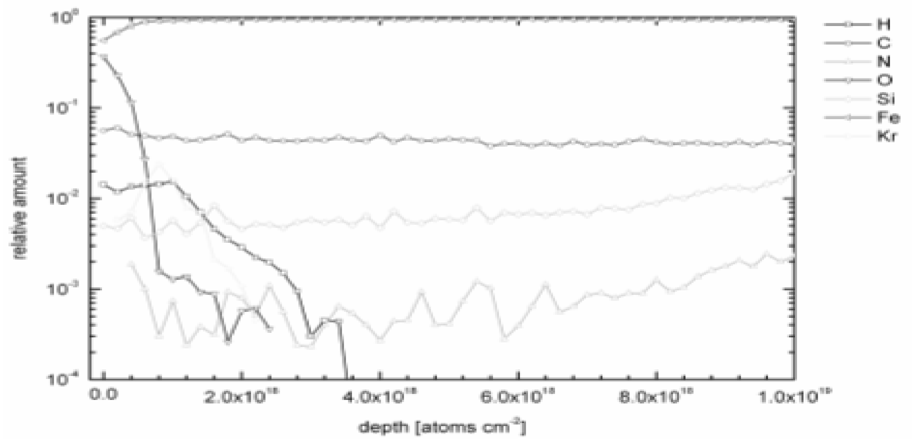

Figure 13. Results ERDA analysis for sample – Kr^{8+}

4. CONCLUSIONS

On the basis of presented results, several conclusions can be drawn:

1. Ion implantation of Kr can decrease surface nano roughness of cold working steel 100Cr6, however results depend on the Kr ion energy.
2. ERDA profile shows significant differences in nano surface zone composition, depending on the applied type of implanted Kr ion.
3. Friction coefficient depends on applied load and significantly depends on type of applied Kr ions and their energy.
4. Obtained results of nanohardness measurements show that the depth of the modified layer is in a range of 30nm which is in accordance with the results of the Monte Carlo simulation.

Based on previous statements it can be concluded that nanomodifications of surface layers can be successfully induced by implantation of Kr ions, and this paper is among the first in the world that address this field. Monte Carlo simulation can be of great help in order to obtain needed ion energy in function of the base material.

Ion implantation is very important and promising technique for surface nanomodification, especially in case of ultra precision tools where depth of modification should be in range of 30-100nm. Important advantage of this technique is the fact that surface dimension is not changed after implantation. This is especially important for ultra precision tools where the subsequent dimension correction would be extremely difficult.

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