

We are IntechOpen, the world's leading publisher of Open Access books Built by scientists, for scientists

4,800

Open access books available

122,000

International authors and editors

135M

Downloads

Our authors are among the

154

Countries delivered to

TOP 1%

most cited scientists

12.2%

Contributors from top 500 universities



WEB OF SCIENCE™

Selection of our books indexed in the Book Citation Index
in Web of Science™ Core Collection (BKCI)

Interested in publishing with us?
Contact book.department@intechopen.com

Numbers displayed above are based on latest data collected.
For more information visit www.intechopen.com



Introductory Chapter: Preferential Sputtering and Oxidation of Nb-Ta Single Crystals Studied by LEIS

Vadim Glebovsky

Additional information is available at the end of the chapter

<http://dx.doi.org/10.5772/65016>

Metal alloys—macroscopically homogeneous metallic materials consist of a mixture of two or more chemical elements with a predominance of metal components. The alloys are one of the major structural materials. The technique uses more than five or six thousand alloys. The solid-state alloys can be homogeneous or heterogeneous. The alloys may be presented as interstitial solid solutions, substitution solid solutions, chemical compounds, and simple substances as crystallites. The properties of alloys are completely determined by their crystal structure or phase microstructure. The alloys exhibit metallic properties, such as electrical conductivity, thermal conductivity, metallic luster, and ductility. Such a detailed list of seemingly simple things would be surprising if in every word it has not been hidden in the centuries of research, mistakes, achievements, and discoveries. If desired, anybody could write an exciting-romantic-adventure novel, describing the history of the particular alloys and their role in human life.

Until now, the term “metal” was more or less associated with the term “crystal,” whose atoms are arranged in space in a strictly orderly fashion. In the middle of the last century, scientists discovered the metal alloys having no crystalline structures, that is, amorphous metal alloys with a disordered arrangement of atoms in space. Metals and alloys with disordered arrangement of atoms became known as amorphous metal glasses, paying tribute to the analogy that exists between the disordered structure of a metal alloy and an inorganic glass. Discovering amorphous metals made a great contribution to the science of metals, significantly changing our ideas about them. It was found that amorphous metals are very different in their properties from the metal crystals, which are characterized by an ordered arrangement of atoms. Formation of an amorphous structure of metals and alloys lead to fundamental changes in the magnetic, electrical, mechanical, and even superconducting properties. Some of them were very interesting both for science and for application. The emergence of amorphous alloys—it is not the single result of scientific research being conducted in materials

science and physics of metals. Virtually every group of metal alloys, such as iron-based or titanium-based alloys, have a long and interesting history.

In general, metallic alloys are the interdisciplinary subject or even an area that cover physics, chemistry, material science, metallurgy, crystallography, *etc.* This book, which you, dear readers, are holding in your hands or watching on your PC monitor, is devoted to this old/new subject—the metallic alloys. The primary goal of this book is to provide coverage of advanced topics and trends of R&D of metallic alloys. The chapters of this book are contributed by the respected and well-known researchers in this area. They have presented the up-to-date developments of the metallic alloys technologies. The book consists of 10 chapters divided into two sections of the metallic alloys including the studies of amorphous and nanoalloys, modeling of disordered metallic alloys, superconducting alloys, differential speed rolling, meta-magnetic Heusler alloys, *etc.* We hope that you, dear readers, will find this book interesting and helpful for your work and studies. If so, this could be the best pleasure and reward for us.

As scientific editor of this book, I had to read all chapters and more than once, especially if the chapter does not meet the standards adopted in the publishing house. In particular, this could be due to a deviation from the scope of the manuscript or its translation, scientific content or quality of the so-called similarity (plagiarism) of a manuscript. I was a bit lucky—the authors of submitted manuscripts were, as a rule, consistent with accepted standards, although there were also some deviations. So, part of the manuscripts had an increased volume (text, figures) that was solved through negotiations between publishers and authors. A CrossCheck program, through which the manuscripts are analyzed, records all matches with publications in all editions, and within a reasonable time. In our case, there are no borrowing from the “other people’s” publications (which is a real plagiarism), but only self-citation, when the manuscript contains pieces from own articles. Sometimes the index of self-citation is very rude, and the authors have been asked to correct the situation. Once the authors did not agree to fix the text and took their manuscript back, which we met with a great regret, because the manuscript contained a very interesting scientific content, and could be, if corrected, one of the best chapters of the book.

I would like to thank all of the authors of this book for their contributed chapters. It is my great pleasure to acknowledge the friendly assistance of Ms Andrea Koric, who continuously showed high professionalism and readiness to support the writing of the book from its very beginning to the final format. I also would like to express my deep thanks to my lovely daughter Nastya and my son Kirill, for their patience and love, throughout all my years in science.

At this point, I would like to finish the formal part of my “Introductory chapter: Preferential sputtering and oxidation of Nb-Ta single crystals studied by LEIS.” and switch to my research contributions to metal alloys. In different periods of my scientific life, I had to deal with a variety of metals and alloys: the iron-carbon alloys and different steels, many alloys for thin-film metallization based on high-purity single-crystalline refractory metals Mo, W, Nb, Ta, Ti, the systems Pt/Pd and W/Ti for microelectronics, different alloys for bio-implants, single-crystalline alloys of Nb-Ta. About the preferential sputtering and oxidation of these alloys studied by low-energy ion scattering, I would like to tell in the Introductory chapter.

Currently, Nb-Ta alloys are used in many fields of science and technology: in the electric appliances and electronics, in the chemical industry for the manufacture of chemical apparatuses, in the rocket technology for the manufacture of the nozzle heads, and others. Nb superconducting alloys are used in heavy duty atomic accelerators for manufacturing windings magnets for hot plasma reflectors, lasers, and other nuclear power plants. It is also known the use of the alloys in aviation technology for manufacturing uncooled turbine blades in jet engines, and others. Nb-/Ta-based alloys currently provide performance products at temperatures up to 1300°C and based on Ta up to 1700°C. Despite the higher melting temperature of alloys based on Ta, they are less common than Nb-based alloys. There are several reasons for that; the main is the scarcity and high price of Ta. Therefore, in recent years, began to attract the attention to new ideas: In the manufacture of these elemental metals, they are not separate; indeed, why separate them, if they are always related to each other in nature and, therefore, supplement each other in alloys excellently.

Ta has a unique feature—it is the only metal that has a biological compatibility with a living tissue. Metal, named after the mythological martyr, has an interesting mission to the mankind—it came to the aid of man, his living tissues. In reconstructive surgery and neurosurgery, Ta began to be used during World War II: The replacement of the damaged parts of the skull, bound broken bones, replacing the small bones with the wire and the metal strips. Ta yarn and mesh used for the replacement of muscle tissues, and as a basis for the growth of new tissues. A metal mesh is used to reinforce the walls of the abdominal cavity, with the help of a thin Ta wire to stitch tendons and damaged nerves. A similar property—a biocompatibility—is a characteristic of Ta-Nb alloys. A lower density compared with Ta alloys that makes them promising. Ta may come to replace stainless steel, gold, and other conventional alloys because, unlike traditional metallic materials for implants, the human body perceives Ta and Nb-Ta alloys, not as a foreign body, but as your own bone. Perhaps, said about Nb-Ta alloys looks more like a hymn to the glory of these metals and their alloys. So let it be—I really admire their unusual physical properties, capabilities, and believe in the enormous potential in the nearest future.

As well known, Nb and Ta, having similar lattice parameters, crystallize in a similar body-centered cubic lattice. Both metals have similar chemical and physical properties. Thus, the Nb-Ta system should have a continuous range of the substitutional solid solutions (alloys) [1]. Moreover, the pure Nb and Ta and their alloys can be produced as single crystals with a known crystallography. The physical and chemical interaction of oxygen with Nb and Ta can be studied by the methods of a surface analysis such as low-energy electron diffraction (LEED) or Auger electron spectroscopy (AES), having a larger sensitivity depth than LEIS [2–13]. The interaction of oxygen with metal surfaces is important in catalysis, corrosion, and growth. A series of single crystals (110) of Nb-Ta alloys has been studied by LEIS for obtaining quantitative information about the single crystals of Nb-Ta alloys during their interaction with oxygen. In this chapter, the results of the LEIS experiments on the single crystals of several Nb-Ta alloys and the elemental Nb and Ta are presented. The contents of Nb and Ta and alloys of the surface oxygen in the upper layer of the surface may be quantified by LEIS, that is a surface analysis technique with extremely high sensitivity and selective atomic layer to the outer surface [14].

When the matrix effects are absent, the composition can be quantified by calibration of surface [15]. As an editor of the book, I would like to present the study of these alloys, which could be a main part of the introductory chapter. The study covers several more or less traditional techniques (levitation melting, EB floating zone growing single crystals of refractory metals, X-ray Laue characterization of single crystals, recrystallization for growing massive single crystals of alloys, elemental characterization by ICP MS, and others) and UHV techniques for studying upper layers of single crystals (LEIS, LEED, SIMS). By techniques used as well by the aims and results, this study is also traditional. A part of experiments is done in ISSP RAS, Chernogolovka, another part of the study is done together with Prof. Hidde Brongersma in TUE, Eindhoven.

1. Experimental

The alloys of Nb and Ta are obtained by mixing the pure elemental powders in a desired ratio by using a high-frequency levitation melting. This method is crucible less—the metal sample melts in an electromagnetic field formed by a conic inductor. The radio-frequency electromagnetic field provides a uniform mixing of both metals in the liquid state [16]. To form cylindrical cast rods, the melt is cast into a cylindrical water-cooled copper mold. Single crystals of these alloys are grown by electron beam floating zone melting which provides refining material together with a uniform distribution of both elements in the volume [17]. Single crystals of both metals and three Nb-Ta alloys are grown with a growth rate of 3 mm min^{-1} using the specially prepared single-crystalline seeds of three main crystallographic orientations—(111), (110), and (100). For this part of the study, the discs of the (110) plane index are cut off by electro-erosion and then mechanically and chemically polished. X-ray Laue back reflection is used to a crystallographic check of the as grown crystals and final discs. It was discovered that $\text{Nb}_{0.75}\text{Ta}_{0.25}$, $\text{Nb}_{0.5}\text{Ta}_{0.5}$, and $\text{Nb}_{0.25}\text{Ta}_{0.75}$ alloys could not be grown directly from the melt as crystals. In order to grow crystals of these alloys, recrystallization is used which consists of a strain deformation followed by a high-temperature annealing (up to the melting temperature of alloys). For this study, the following groups of single crystals are grown in different volume composition: Nb, $\text{Nb}_{0.75}\text{Ta}_{0.25}$, $\text{Nb}_{0.5}\text{Ta}_{0.5}$, $\text{Nb}_{0.25}\text{Ta}_{0.75}$, and Ta. Rutherford backscattering spectroscopy (RBS) is used to check the composition of the volume. Contents of both metals in alloys are analyzed also by ICP MS.

2. SIMS and SNMS experiments

Before LEIS experiments, the alloys are studied by SIMS and SNMS. These measurements are made by a 200 quadrupole mass spectrometer Leybold SSM. The base pressure in the system is in a low range of 10^{-10} mbar. The Ar^+ primary beam with energy of 5 keV is used. Static SIMS spectra of the surface are recorded with a current density of 50 cm^{-2} , and with a typical acquisition time of 200 s. This leads to a total dose of 5×10^{13} ions cm^{-2} , which is a static limit. Bulk analyses by SIMS and SNMS are performed with a higher current density of 5 mA cm^{-2} ,

and an acquisition time of 1 h to improve a signal-to-noise ratio. The SNMS is emitted by ionized forms of a post-60 eV electron beam. The samples are of the (100) surface orientation, in order to eliminate the effect of differences in density between various lattice planes [18]. Firstly, it is shown that prolonged sputtering is important for obtaining meaningful SIMS spectra with dimers and trimers of Nb. Next, the SIMS and SNMS spectra of Nb, Ta, and NbTa alloys are compared. Positive SIMS spectra of the as grown Nb single crystal in the mass range from 75 to 315 atomic mass units (amu) are measured. Since the spectrum is measured in a static mode, it shows the composition of the surface. The Nb⁺ peak is at 93 amu, and it dominates the spectrum. Contaminants can be seen in the form of cluster ions such as NbC⁺ (105 amu), NbN⁺ (107 amu), NbO⁺ (109 amu), NbF⁺ (112 amu), and NbO₂⁺ (125 amu). The presence of hydrogen, which is easily dissolved by these metals, is represented by NbH⁺ peak at 94 amu. In the higher mass range, a small surface contamination by Ta is visible in the peaks of Ta⁺ (181), TaO⁺ (197 amu), and TaO₂⁺ (213 amu). Of some interest is a dimer Nb₂⁺. However, this peak has a low intensity, because it is very sensitive to the surface cleanliness. In a spectrum of the same Nb crystal after 30 min of sputter with 5 keV Ar⁺ ions at a density of 5 μA cm⁻², there are high intensities of Nb₂⁺ and Nb₃⁺ clusters, while clusters which are typical of impurities have a much lower intensity than in the first spectrum (without long sputtering). During etching, various secondary ion signals are recorded. Several characteristic intensity ratios are registered. Carbon and oxygen are removed within the first 5 min of the etching process, which corresponds to a removal of several tens of atomic layers. Simultaneously with the removal of impurities, dimers increase the intensity of Nb on the order of magnitude, and thus, Nb trimer peak appears. Small peaks of NbC⁺ and NbO⁺ remain but correspond to carbon and oxygen concentrations below the limit of detection of Auger electron spectroscopy. The conclusion is made that the long-term etching to achieve sputter balance is essential to obtain stable spectra, which are really representative of the bulk composition.

3. LEIS experiments

The unique properties of LEIS in combination with new instrumental developments allow conducting research in emerging areas of science and technology. **Figure 1** shows some of the characteristics of LEIS in comparison with such widely used analytical techniques such as Auger electrons spectroscopy (AES), X-ray photoelectron spectroscopy (XPS), secondary ion mass spectroscopy (SIMS). It is clear that none of these techniques has any such high depth sensitivity as LEIS to the topmost atomic layer. The treatment of information obtained by means of analytical methods for studying the surface is quite complicated. While SIMS method has the highest sensitivity to alkali metals, LEIS is far more sensitive to the noble metals and especially to metals with high atomic masses [19].

A target surface in LEIS is irradiated with a monoenergetic beam of inert gas ions with energy in the range of 1–5 keV. Upon reaching the surface of the target, an ion undergoes one or more collisions with the target atoms. The ion scattering spectroscopy investigates the energy distribution of the primary ions, backscattered in a vacuum. The de Broglie wavelength for ions with energy of 1 keV is very small compared with the interatomic distances on the surface.

Thus, in contrast to the scattering of electrons and phonons, the majority of ion scattering phenomena can be quite accurately described by the methods of classical mechanics. The ions are scattered by the Coulomb interaction between the (shielded) nucleus of the ion and atom. Under normal experimental conditions, this interaction is important only for distances of less than 0.05 nm. This is a good approximation for the assumption that at any given moment an ion interacts with a single atom. Since the time of interaction ($\sim 10^{-15}$ s) is very small compared with the characteristic time for the phonons ($\sim 10^{-12}$ to 10^{-13} s), the target atom can be considered as a free atom. In the process of scattering, an ion loses some of its kinetic energy. Energy losses can also be accurately calculated in the approximation of elastic scattering. LEIS experiments are conducted using the scattering apparatus ions (**Figure 2**). Primary ions are formed in the ion source and directed perpendicular to the target surface. Ions are dispersed to 1440 target atoms and energy is analyzed by a cylindrical mirror analyzer. Using very pure ion beams is essential to obtain a low level of background spectra. The nominal base pressure in the vessel is in a low range of 10^{-10} mbar and can be controlled by a quadrupole mass spectrometer. The device is equipped with a source of ions for sputtering at a grazing angle of 15° .

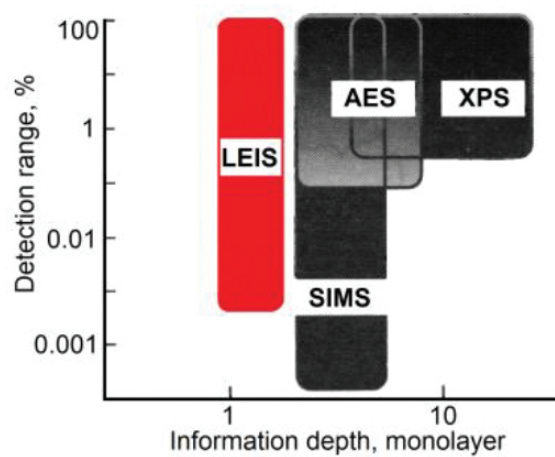


Figure 1. Comparison of LEIS with SIMS, XPS, AES.

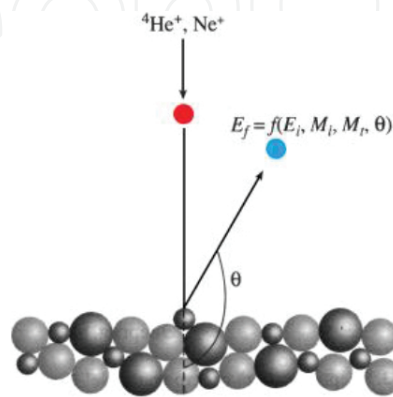


Figure 2. LEIS experiment.

4. Adsorption of oxygen and sputtering

Surfaces of Nb-Ta alloys are purified by the Ar ion-sputtering cycles at room temperature and annealed at 800 K. This temperature is too low to remove all the defects. In addition, it is impossible to remove all the oxygen in this way, but it is effective to remove surface contaminants (carbon, nitrogen, hydrogen). For achieving atomically clean surfaces, there are necessary to have the annealing temperatures above 2000 K; thus, it seems our annealing is not yet available. Oxygen (99.995%) is supplied to a vessel with a dose of 30–50 L (Langmuir), which is high enough to saturate the surface. **Figure 3** shows the typical LEIS spectra of clean and oxygen-covered Nb-Ta alloy. By coating the surface of Nb-Ta with oxygen, Nb and Ta peaks are screened, and consequently, the Nb and Ta intensities decrease. The following procedure allowed us to study this effect in more detail. The sample is first saturated with oxygen. Then oxygen is evacuated, and LEIS spectra measured with 1.5 keV $^4\text{He}^+$ primary ion beam which also provides a slower removal of oxygen from the surface by sputtering. The procedure is repeated for a reproducibility check. Thus, LEIS spectra of alloys and metals with different oxygen coverage can be obtained. The intensities for Nb and Ta depend linearly on the intensity of oxygen. Some changes in the primary ion beam time can be corrected by calibration against a clean Cu surface. Thus, the final composition effects on secondary electron emission and the effective current target can be avoided.

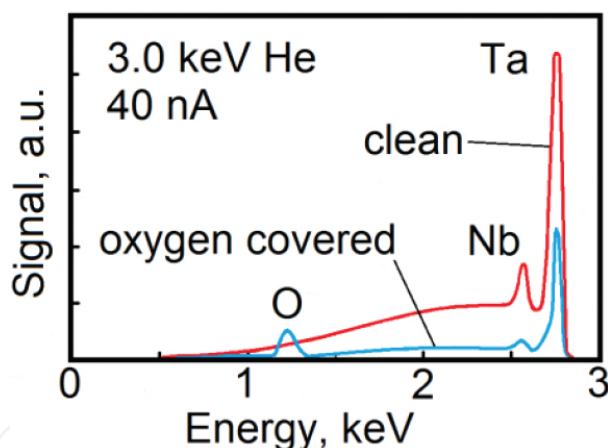


Figure 3. Typical LEIS spectra of the oxygen-coated and clean surfaces of (110)Nb_{0.75}Ta_{0.25}. The ion $^4\text{He}^+$ with energy 3.0 keV, output current of 40 nA. To reduce measurement time, the oxygen-coated sample is only measured in the range of interest.

5. Quantification of Nb and Ta at the surface

In **Figure 4**, the linear dependence shows that there are no matrix effects for these ion-atom combinations. Removing oxygen by sputtering, apparently does not affect the scattering process and ion fractions of the adjacent atoms of Nb and Ta. Only more atoms of Nb and Ta are exposed to the primary ion beam, which corresponds to an increase in the Nb and Ta

signals. Such behavior differs from that of the secondary ion mass spectrometry (SIMS), where part of the ion-sputtered particles changes drastically by the presence of oxygen. To obtain Nb and Ta signals for Nb_xTa_{1-x} alloys without oxygen, the lines in **Figure 4** are extrapolated to zero oxygen coverage. It is interesting that a linear relationship has been obtained when plotting the extrapolated Ta signals as a function of the corresponding extrapolated Nb signals, if no matrix effects present in these LEIS experiments [19, 20]. Results in **Figure 5** show that this prediction is performed within an experimental error. Deviations of approximately 15% in the linear relationship between Nb and Ta signals can be result of several reasons. Positioning and focusing system should be made individually for every sample. The signals are calibrated with a standard Cu specimen. Both dimensions have errors of a few percent. The bulk material is of very high purity; however, adsorption and segregation can change the situation and increase the content of impurities on the surface.

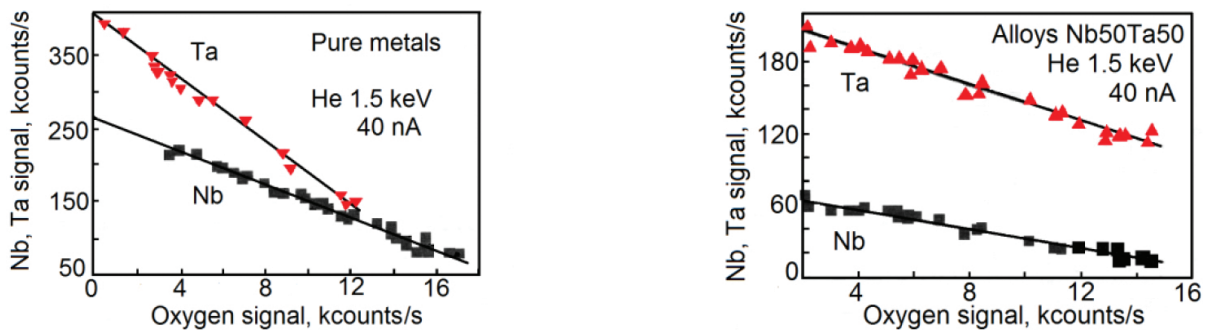


Figure 4. Nb and Ta signals vs. oxygen signals for LEIS on pure metals and $Nb_{0.5}Ta_{0.5}$.

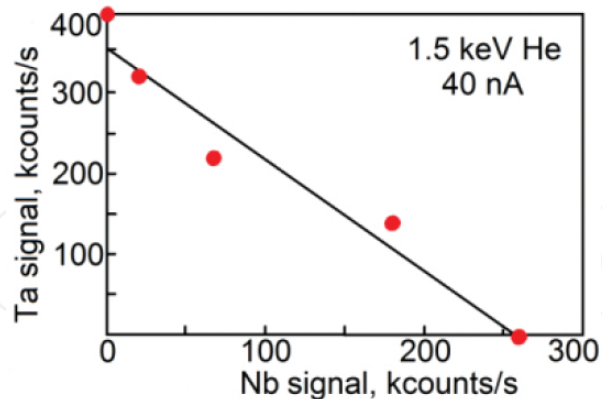


Figure 5. The peak intensity Ta vs. Nb alloys for Nb_xTa_{1-x} system without oxygen.

As for carbon, with a low sensitivity in LEIS, it is difficult, if possible, to detect. Different patterns could have different contents of impurity atoms on the surface. Because of the low temperatures of sputtering and annealing, the surface of different samples could not be a perfect (110) plane. With changing the structure of the sample surface, Nb and Ta densities become lower than that of a higher density packaged (110) plane. Determination of the peak

intensity of Nb in a LEIS spectrum is not a simple task for such alloys because Nb peak overlaps Ta peak of a low-energy tail. A special oven has been used for high-temperature (2000°C) annealing the samples in the preparation chamber. A linear curve in **Figure 5** can be used to calculate the surface composition for clean Nb_xTa_{1-x} alloys, since the signals for both metals are proportional to their content on the surface. The experimental data are plotted and make a straight line through the experimental points to the beginning of the graph coordinates, taking the intersection with a linear curve. The Nb and Ta surface contents are found by dividing the transferred Nb and Ta signals by the signals of pure Nb and Ta, respectively. The surface contents of the alloys calculated in the described way are shown in **Figure 6**. The surface of samples is clearly enriched in Ta. These alloys have very high melting temperatures (2690–3270 K); thus, thermally activated surface segregation can be neglected at room temperature. Nb-Ta alloys are the ideal systems for an experimental determination of the role of the mass difference on the preferential sputtering of both metals from the matrix. Sigmund's theory [20] gives the ratio R of the sputter yield Y_{Nb} of Nb to that of Ta Y_{Ta} : where N_{Nb} and N_{Ta} are the atomic concentrations (number of atoms per unit volume), M_{Nb} and M_{Ta} , the atomic masses, and U_{Nb} and U_{Ta} , the surface binding energies of Nb and Ta, respectively. The exponent m , which is currently expected to about 1/6, is a parameter characteristic of the interaction potential.

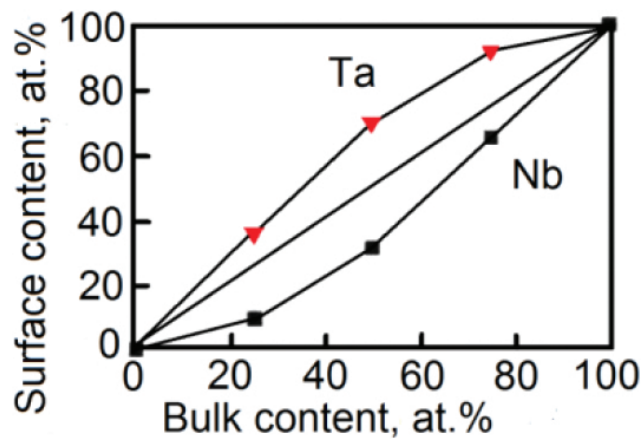


Figure 6. The concentrations of Nb(Ta) on the surface against Nb(Ta) in the volume (in at.%). Sigmund's model (central line) for preferential sputtering is shown for comparison.

$$R = Y_{Nb} / Y_{Ta} = N_{Nb} / N_{Ta} (M_{Ta} / M_{Nb})^{2m} (U_{Ta} / U_{Nb})^{1-2m} \quad (1)$$

The ratio of the surface binding energies of Ta and Nb is estimated to be equal to the ratio enthalpies of evaporation of these elements (1,09) [21]. Since sputtering is mainly limited to atoms by the outermost layer, the preferential sputtering of Nb should lead to enrichment in Ta in the upper layer with a factor of 1.3. However, the observed enrichment is even higher than predictions based on the preferential sputtering (**Figure 6**). Since our setup does not allow for removal of oxygen when heated, it is likely that oxygen-induced segregation in combination with a primary sputter can be a reason for the observed effect.

6. Quantification of oxygen

The quantification of the oxygen signal may be done using a calibration with respect to the surface of Ni(100)–O_c(2 × 2). As known, this is a very stable structure that is obtained when the surface is saturated with oxygen, has oxygen coverage of half of a monolayer, and corresponds to 8×10^{14} atoms of oxygen per 1 cm^{-2} . For calibrations, oxygen adsorption on Ni(100) is studied by LEIS under the same conditions as in the experiments with Nb-Ta alloys (Figure 7), again using signal of pure Cu for normalization.

In Figure 7, the maximum coverage of oxygen on Ni(100) corresponds to the density of oxygen atoms 8×10^{14} atoms cm^{-2} and gives the oxygen signal of 7.3×10^3 counts s^{-1} . A linear decrease of the Ni signal with increasing the oxygen signal demonstrates the lack of matrix effects. The quantification of the maximum oxygen concentration in Nb-Ta samples using this calibration is possible. Dividing the oxygen density by the metal density, that is 13.0×10^{14} and 12.9×10^{14} atoms cm^{-2} for Nb(110) and Ta(110), respectively, provides values of an oxygen coverage. The results are shown in Table 1.

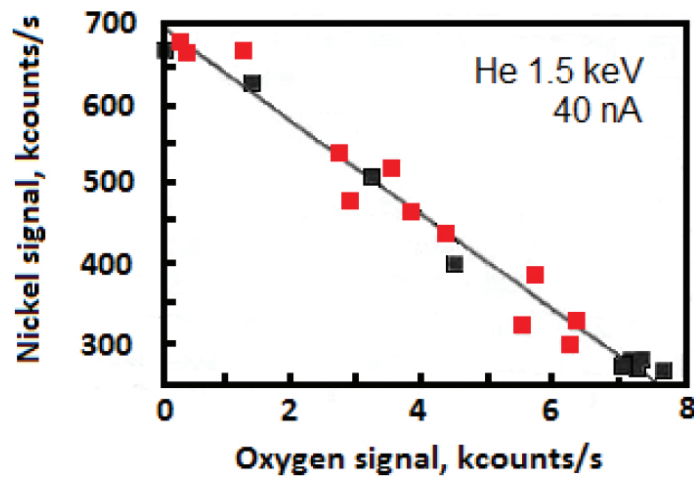


Figure 7. Ni peak intensity as compared to the O peak intensity to the surface of the Ni(100). Various oxygen coverages obtained by sputter (red) and by monitoring the oxygen exposure (black).

Sample	Surface composition		Maximum oxygen density (10^{15} at cm^{-2})	Maximum oxygen/metal ratio
	%Nb	%Ta		
Nb	100	–	1.82	1.41
Nb _{0.75} Ta _{0.25}	65	35	1.72	1.33
Nb _{0.5} Ta _{0.5}	31	69	1.51	1.17
Nb _{0.25} Ta _{0.75}	9	91	1.43	1.10
Ta	–	100	1.30	1.00

Table 1. Quantitative composition of the (110)Nb-Ta alloys by LEIS.

An oxide growth on Ta (110) and Nb (110) is described by the formation TaO(111) [3] and NbO (111) [8], respectively. The oxygen/metal ratio of 1.0 that we get with LEIS for Ta(110) is very close to these studies. For Nb(110), however, it is found the oxygen/metal ratio of 1.4, which is higher than the value of Ta. The oxygen coverage on Nb is higher because of better shielding of Nb as compared with Ta. Covering and shielding the oxygen atoms on the Nb-Ta alloys increases with increasing the Nb content. For Nb, it is expected that the surface contains more oxygen than Ta surface. Hu et al. [12] reported the existence of two Nb oxides (NbO and Nb₂O₅, detected via XPS) on Nb(110) surface bared to 3000 L of oxygen. Haas et al. [3] observed that the solubility of oxygen in Nb greater than in Ta (4.5% and 3%, respectively). Also, the structure difference between Ta and Nb oxides on the surface can produce differences in the exposed oxygen density on surfaces of Nb or Ta.

Preferential sputtering and oxidation of three single-crystalline (110)Nb_xTa_{1-x} alloy ($x = 0.25, 0.5, 0.75$), together with single crystals of pure Nb and Ta, are studied by LEIS. After sputter cleaning, LEIS showed Ta enrichment on the surface of all NbTa alloys, indicating Nb preferential sputtering. This is in a reasonable agreement with theory. After contact with oxygen, linear relationships between O and Nb and Ta signals indicate that the matrix does not affect the LEIS signals for these systems. LEIS is very useful for collecting quantitative information about the composition of the outer layer of the surface of the alloys. Nb-Ta alloys differ from those in the bulk. The oxygen coverage on NbTa alloys after exposure to oxygen has been determined with an accuracy of about 15% after calibration using a maximum coverage of oxygen in the known Ni system (100)–Oc (2 × 2). The maximum surface oxygen concentration is defined as 13×10^{15} atoms cm⁻² for Ta(110) and 18×10^{15} atoms cm⁻² for Nb(110), which corresponds to the oxygen coverage of 1.0 and 1.4, respectively. The maximum oxygen coverage of the alloys increases with the Nb content.

Author details

Vadim Glebovsky

Address all correspondence to: glebovs@issp.ac.ru

Institute of Solid State Physics RAS, Chernogolovka, Russia

References

- [1] L H Bennett, T W Massalski, B C Giessen. Alloy phase diagrams. North-Holland. Amsterdam (1983).
- [2] J E Boggio, H E Farnsworth. Low-energy electron diffraction and photoelectric study of (110) tantalum as a function of ion bombardment and heat treatment. Surf Sci 1964; 1: 399–406.

- [3] T W Haas, A G Jackson, M P Hooker. Adsorption on niobium (110), tantalum (110), and vanadium (110) surfaces. *J Chem Phys* 1967; 46: 3025–3033.
- [4] B Sewell, D F Mitchell, M Cohen. A kinetic study of the initial oxidation of a Ta(110) surface using oxygen k_{α} X-ray emission. *Surf Sci* 1972; 29: 173–188.
- [5] H H Farrell, H S Isaacs, M Strongin. The interaction of oxygen and nitrogen with the niobium (100) surface: I. Morphology. *Surf Sci* 1973; 38: 31–52.
- [6] H H Farrell, M Strongin. The interaction of oxygen and nitrogen with the niobium (100) surface: II. Reaction kinetics. *Surf Sci* 1973; 38: 18–30.
- [7] R Panlei, M Bujor, J Bardolie. Continuous measurement of surface potential variations during oxygen adsorption on the (100), (110) and (111) faces of niobium using mirror electron microscope. *Surf Sci* 1977; 62: 589–609.
- [8] G Ertl, M Plancher. A statistical model for oxygen adsorption on heterogeneous metal surfaces. *Appl Surf Sci* 1980; 6: 453–463.
- [9] M Grundner, J Halbnitter. On the natural Nb₂O₅ growth on Nb at room temperature. *Surf Sci* 1984; 136: 144–154.
- [10] A V Titov, H Jagodzinski. Structure of O layers on the Ta(100) surface. *Surf Sci* 1985; 152/153: 409–418.
- [11] N Shamir, U Atzmony, J A Schultz, M H Mintz. Hydrogen-oxygen interrelations on a niobium surface. *J Vac Sci Technol A* 1987; 5: 1024.
- [12] Z P Hu, Y P Li, M R Ji, J X Wu, The interaction of oxygen with niobium studied by XPS and UPS—ADS. *Solid State Commun* 1989; 71: 849.
- [13] C Surgers, H V Lohneysen. Effect of oxygen segregation on the surface structure of single-crystalline niobium films on sapphire. *Appl Phys A* 1992; 54: 350–354.
- [14] H H Brongersma, P Groenen, J-P Jacobs. Application of low energy ion scattering to oxidic surfaces. In: *Science of Ceramic Interfaces*, J Novotny (Ed.). Material Science Monographs 8, Elsevier Science B.V., Amsterdam (1994) pp 113–182.
- [15] P A J Ackermans, G C R Krutzen, H H Brongersma. The use of a calibration in low-energy ion scattering: Preferential sputtering and S segregation in CuPd alloys. *Nucl Instr Meth B* 1990; 45: 384–389.
- [16] V G Glebovsky, V T Burtsev. *Levitation Melting of Metals and Alloys*. Metallurgia Publ House. Moscow (1974) 174 p.
- [17] V N Semenov, B B Straumal, V G Glebovsky, W Gust. Preparation of Fe-Si single-crystals and bicrystals for diffusion experiments by the electron-beam floating-zone technique. *J Cryst Growth* 1995; 180: 151.
- [18] H J Borg, J W Niemantsverdrit, H H Brongersma, V G Glebovsky. A SIMS/SNMS study of high-purity Nb_xTa_{1-x} alloys. *J Surf Phys* 1992; 13: 32–36.

- [19] L C A van den Oetelaar, J P Jacobs, M J Mietus, H H Brongersma, V N Semenov, V G Glebovsky Quantitative surface analysis of NbTa alloys by low-energy ion scattering. *Appl Surf Sci* 1993; 70/71: 79–84.
- [20] P Sigmund, M W Sckerl. Momentum asymmetry and the isotope puzzle in sputtering by ion bombardment. *Nucl Instr Meth B* 1993; 82: 242–254.
- [21] F R de Boer, R Boom, W C M Mattens, A R Miedema, A K Niessen. Cohesion and structure. In: *Cohesion in metals: transition metal alloys*, F R de Boer, D G Pettifor (Eds.). North Holland. Amsterdam 36 (1988) pp 385 and 539.

IntechOpen

