
ARTICLES

Optimization of Material Analysis Conditions for Laser-Assisted Atom Probe Tomography Characterization

O. A. Raznitsyn^{a, b, *}, A. A. Lukyanchuk^{a, b}, A. S. Shutov^{a, b}, S. V. Rogozhkin^{a, b}, and A. A. Aleev^{a, b}

^a*Institute for Theoretical and Experimental Physics named by A.I. Alikhanov, National Research Centre “Kurchatov Institute”
Moscow, 117218 Russia*

^b*National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Moscow, 115409 Russia*

**e-mail: Oleg.Raznitsyn@itep.ru*

Received February 3, 2017; in final form, February 9, 2017

Abstract—Atom probe tomography (APT) analysis with a femtosecond laser is one of the most promising techniques to characterize local variations of chemical distribution in materials with subnanometer spatial resolution. In this study, the effect of APT analysis conditions on the accuracy of data recovery for chromium steels and Fe-Cr alloys is investigated. Dependences of mass resolving power and the detected composition of chromium as a function of laser power, specimen geometry, and evaporation rate are presented.

Keywords: atom probe tomography, time-of-flight mass spectrometer, laser evaporation

DOI: 10.1134/S1061934817140118

INTRODUCTION

Atom probe tomography (APT) in recent years has become widespread in materials science because of a number of advantages over other methods of ultramicroscopy. This technique uses the principle of a projection microscope, similar to field ion microscopy, which ensures the reconstruction of the spatial distribution of atoms in material with a resolution of approximately 3 Å. The implementation of time-of-flight mass spectrometry for each evaporated ion of a material enables the determination of its chemical nature. As a result, this technique allows to reconstruct three-dimensional atom maps of analyzed specimens with the chemical identification of each individual atom [1], which is the main advantage over other methods of nanoscale analysis.

An APPLE-3D atom probe tomograph with laser evaporation was recently developed and launched at the Institute for Theoretical and Experimental Physics named by A.I. Alikhanov of National Research Centre “Kurchatov Institute” [2]. The implementation of APT with laser evaporation not only improves the quality of the analysis of dielectric inclusions in conducting materials, but will also ensure the investigation of semiconductors and dielectrics. An important element of developing a new device is testing the analysis of materials in which parameters yielding the most reliable results are selected. In this paper the effect of the APT analysis parameters of the APPLE-3D on the accuracy of materials reconstruction based on iron and chromium alloys was demonstrated.

PARAMETERS OF TOMOGRAPHIC ATOM PROBE ANALYSIS WITH LASER EVAPORATION OF ATOMS

Laser evaporation in atom probe tomography has been developed in the world since 1970 [3]. With the advent of laser systems with pulse duration of less than a nanosecond, capable of keeping beam parameters with high accuracy for a long time in the mid-2000s, the active use of laser evaporation in APT was began [4]. Activation of the process of field evaporation by means of a laser is carried out in the following way: the laser pulse transmits energy through the electron subsystem to the surface layer of the specimen, so that the surface atoms can overcome with some probability the potential barrier holding them, lowered by voltage E applied to the specimen [4]. The probability of the evaporation of an ion P in this case is described by the Arrhenius dependence

$$P \sim \exp\left(\frac{-Q(E)}{k_B T}\right), \quad (1)$$

where $Q(E)$ is the activation energy, k_B is the Boltzmann constant, and T is the local temperature of the specimen in the ion evaporation region.

As a specimen tip heated by a laser cools down not instantaneously, but for some period of time (~ 10 ns) [4], it is possible that the ions evaporate within times exceeding the pulse length of the laser. This effect causes the broadening of the peaks in the mass spectrum, which in turn lowers the accuracy of chemical identification of detected atoms.

Table 1. Chemical composition (wt %) of the materials under study and their heat treatment

Brief name of the alloy	Cr	C	Mn	V	W	Ni	N	Si	Ti	Heat treatment
Fe-12Cr	11.17	0.15	0.74	0.25	1.13	0.03	0.04	0.33	-	Quenching at 1100°C for 40 min + drawing back at 720°C for 3 h
Fe-14Cr (+0.3% Y ₂ O ₃)	13.5				2				0.3	Mechanical alloying of matrix steel powders, Ti, and Y ₂ O ₃ in an attritor mill under H ₂ ; degassing in a vacuum at 400°C for 4 h; hot isostatic pressing at 1150°C and 100 MPa for 2.5 h
Fe-22Cr	20.10 ± 0.02									Quenched alloy (purity 99.95%), followed by homogenization at 850°C

Variation of the APT analysis parameters may result in avoiding or reducing this peak broadening caused by heating of the specimen. In the process of APT analysis, variation of some conditions also ensures the achievement of the best reliability and increase in the amount of data obtained. The main variable conditions are the magnitude of pulse action (in our case, the laser power), the evaporation rate at a fixed frequency of pulse actions (which can vary because of a change in the voltage applied to the specimen), and the base temperature of the specimen (the temperature of the specimen at some distance from its tip, supported by a cryosystem and controlled by temperature sensors). In the case of laser evaporation, the shape of the specimen is added to the listed parameters, as the rate of heat removal from the tip depends on it.

In the present work, qualitative dependences of mass resolution, sensitivity, and accuracy of the determination of concentrations on the applied laser power, the shape of the specimen, and the intensity of evaporation are obtained. These dependences significantly accelerate the selection of analysis parameters by choosing certain conditions that correspond to the required accuracy of data recovery when analyzing materials using an atom probe tomograph.

EXPERIMENTAL

APPLE-3D atom probe tomograph. In a prototype APPLE-3D of the APT with laser evaporation [2], the specimen and detector are arranged opposite to each other with a distance between them of approximately 183 mm. The position-sensitive detector based on delay lines the DLD120 (Roentdek) has a diameter of the sensitive region of 120 mm and a detection efficiency of 60%. The used TETA-25ST laser system (Avesta Ltd.) is based on an ytterbium fiber laser generating pulses of 300 fs with a maximum frequency of 50 kHz. The output harmonics have a wavelength of 515 nm, the APT analysis parameters used in the present work are optimized with respect to this wavelength. The beam is focused on the specimen using a lens with a focal length of 25 cm. The used power range is from

1 to 100 mW. Data can be collected at temperatures of 20–100 K. The evaporation rate can be up to 10³ events/s.

The data were reconstructed using special software developed in the Institute for Theoretical and Experimental Physics of National Research Center “Kurchatov Institute.”

Materials used. At present, steels based on Fe-Cr alloy are widely used because of their high corrosion resistance, strength, and high hardness. A wide range of studies of these materials with different concentrations of chromium has been conducted in the laboratory of atomic-scale studies of condensed matter of the Institute of Theoretical and Experimental Physics: Eurofer 97 [5] and ODS Eurofer [6] with 9% of Cr, Rusfer EK-181 with 12% of Cr [7], ODS 14Cr with 13.5% of Cr [8], model alloys of Fe–22% of Cr [9], etc. Studies of these materials were carried out using an ECOTAP (CAMECA) atom probe tomograph with evaporation of atoms by a pulsed electric field. Significant data sets for these materials have been obtained both in the initial state and after thermal treatments or irradiation. To test the procedure of atom probe tomography with laser evaporation, we selected 12% chromium steel Fe-12Cr, 14% chromium oxide dispersion strengthened Fe-14Cr steel, and model Fe-22Cr alloy as specimens in the present work. The dependences of the mass resolution on the shape of the specimens and on the laser power are demonstrated using Fe-12Cr and Fe-14Cr materials. The model high-purity homogenized Fe-22Cr alloy was selected to analyze the accuracy of determination of concentration by varying such experimental conditions as laser power and evaporation rate. Compositions and information on specimen preparation and finishing heat treatment are given in Table 1. It should be noted that in order to avoid errors in determination of concentrations, a surface layer 5–10 nm in thickness, which may contain oxides formed during the preparation and transportation of specimens, was excluded from the analysis.

Specimen preparation. The specimens analyzed in this work were prepared as follows. As-received speci-

mens of the material were cut by electroerosion cutting into rods $0.3 \times 0.3 \times 10$ mm in size. Then the tops of the specimens were conically shaped by electrochemical etching in an electrolyte solution consisting of 5% of perchloric acid in ethylene glycol monobutyl ether. Further, an additional electrochemical polishing of the specimen was carried out in a drop of the above solution under an optical microscope with magnification $100\times$. This procedure is the conventional method for specimen preparation for APT and is widely used throughout the world for the preparation of specimens of conductive materials [1, 2, 4]. The shape of the specimens was monitored with a JEOL JEM1200 EX transmission electron microscope in our study.

RESULTS AND DISCUSSION

Dependence of mass resolution and sensitivity on the specimen shape. As already noted, when laser evaporation was used in APT, the shape of the specimen could be considered a variable analysis condition, since the use of specimens with different geometries can significantly affect not only the volume of the data obtained, but also other characteristics of the accuracy of their recovery [10].

In the APT analysis of a specimen, any type of ion evaporation increases its radius at the tip and, correspondingly, decreases electric field strength in the evaporation region, which is compensated by an increase in voltage applied to the specimen. Increasing voltage increases the energy of the ion and shortens its flight time to the detector. This, correspondingly, affects the accuracy of the determination of the time of flight and worsens mass resolution. Thus, the use of specimens with a small radius at the tip is more preferable for obtaining the highest resolution by mass and sensitivity (capability of resolving peaks in the mass spectrum). Note that the characteristic tip radii in APT studies are from 10 to 100 nm.

One of features of laser evaporation in APT is that, at the tip radii above 40 nm, a noticeable increase in mass resolution may occur as the specimen is evaporated. This effect is due to the manifestation of uneven heating because of the nonuniform absorption of laser energy [11]. When a laser pulse is applied to conical specimens with sizes smaller than the wavelength, there are zones with different peak temperatures. When the zone with the highest temperature is located on the top of the specimen, the tip is cooled more rapidly because of a larger temperature gradient, which favorably affects mass resolution. According to calculations [11] for a 515-nm harmonic, this occurs just at a radius of ~ 60 nm.

To demonstrate these effects, the data obtained in the analysis of Fe-12Cr and Fe-14Cr materials were used. A specimen of the Fe-12Cr alloy had a small initial tip radius (~ 10 nm), and a specimen of Fe-14Cr had a larger initial tip radius (~ 40 nm). The mass spec-

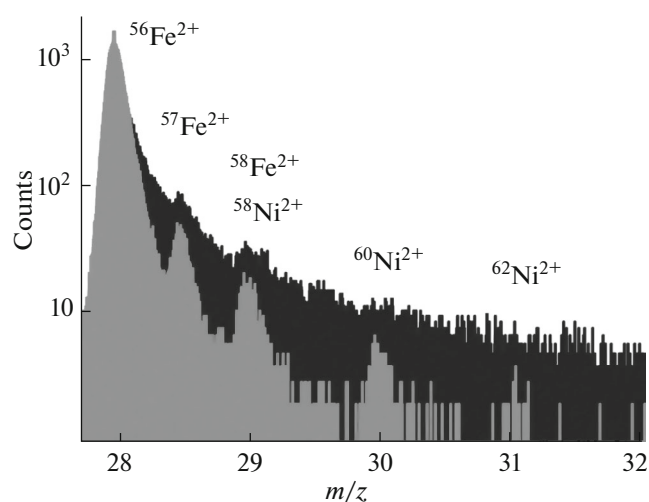


Fig. 1. Mass spectra of Fe-12Cr steel obtained at the beginning of the atom probe tomography study at a specimen tip radius of ≈ 10 nm (gray color) and at the end of the study at a tip radius of ≈ 40 nm (black color) at a fixed angle at the tip.

tra obtained at the beginning of the APT study of the Fe-12Cr material, when the radius at the tip was ~ 10 nm, and at the end of the APT study, when the radius at the tip increased to 40 nm, are compared in Fig. 1. The temperature of the specimen in the study was set at 40 K; laser power used was 15 mW, frequency of laser action was 25 kHz, and evaporation rate was 100 events/s. The analyzed data statistics was 300000 events per measurement (the above parameters are further assumed standard for data collection, unless otherwise is noted). Specimens were selected so that the whole region of the study had a constant angle at the tip of $\sim 5^\circ$. Specimens with a small initial radius at the tip demonstrated a high sensitivity to elements with low concentration at the beginning of the APT study (Fig. 1). Based on the data in Fig. 1, mass resolution of $M/\Delta M$ of 1/10/50% of the height of the main peak of iron $^{56}\text{Fe}^{2+}$ ($M/\Delta M_{1/10/50\%}$) for the initial data portion with a radius of 10 nm was 56/109/279, and for the data portion with a radius of 40 nm, it was 18/99/355, which no longer permitted the detection of peaks corresponding to iron isotopes $^{57}\text{Fe}^{2+}$ and $^{58}\text{Fe}^{2+}$ and nickel isotopes $^{58}\text{Ni}^{2+}$ and $^{60}\text{Ni}^{2+}$. Note that, in the case considered, mass resolution at the half-height of the main peak was improved. This effect was observed earlier in [10].

The change in mass resolution with an increase in the radius at the tip of the specimen in the range from 40 to 60 nm was considered using another material, Fe-14Cr, because of its higher strength (Fe-12Cr was destroyed when the tip radius increased to 60 nm because of a high potential needed to initiate evaporation). The comparison of mass spectra obtained at the beginning of the APT study of Fe-14Cr, when the

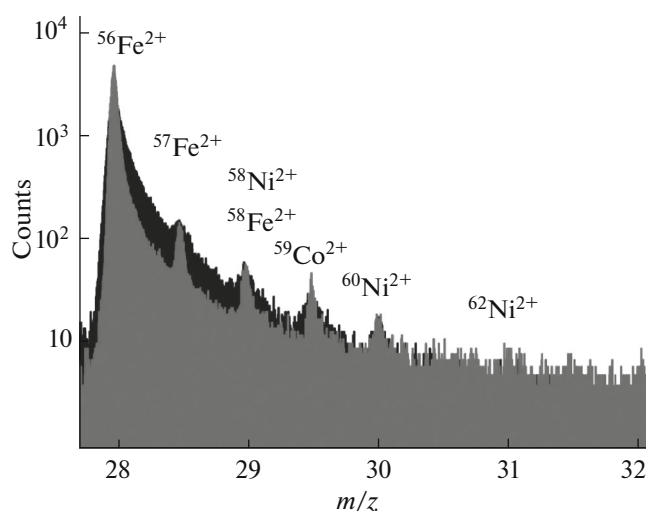


Fig. 2. Mass spectra of Fe-14Cr steel obtained at the beginning of the atom probe tomography study at a specimen tip radius of ≈ 40 nm (black color) and at the end of the study at a tip radius of ≈ 60 nm (grey color) at a fixed angle at the tip.

radius at the tip was ~ 40 nm, and at the end of the APT study, when the radius at the tip increased to 60 nm, is presented in Fig. 2. It can be seen that an increase in the radius at the tip significantly improves mass resolution: it increases by 1% of the main peak of $^{56}\text{Fe}^{2+}$ from 33 to 50; at 10% of the peak height, from 98 to 235; and at half-height of the peak, from 350 to 650. However, despite the high values of mass resolution at the 50 and 10% of the height of the main peak, mass resolution at the height smaller than 1% is not sufficient for the precise chemical identification of peaks of chemical elements standing after the $^{56}\text{Fe}^{2+}$ peak. Identification of such peaks was most accurate for the radius at the tip 10 nm (Fig. 1), where the “tail” of the main iron peak sloped to zero already after 1 amu, enabling a clear observation of peaks after $m/z = 29$, in contrast to the case shown in Fig. 2. Another important parameter of the shape of the specimen was the angle at its tip (we mean the angle in the region of approximately $1\ \mu\text{m}$ from the top of the specimen). The characteristic range of angles at the tip of specimens used was within 10° . With increasing angle at the tip, the rate of heat transfer between the specimen tip and its massive part increased, which accelerated the decrease in surface temperature in the evaporation region and improved mass resolution. The mass spectra obtained in the APT study of specimens with different angles at the tip but with the same radius are compared in Fig. 3. The radius at the tip of a specimen at the beginning of data collection was approximately 30 nm for both cases shown in Fig. 3. It can be seen that the use of a specimen with a large angle at the tip markedly increased mass resolution at 1% of the height of the main peak. In this case, with an increase

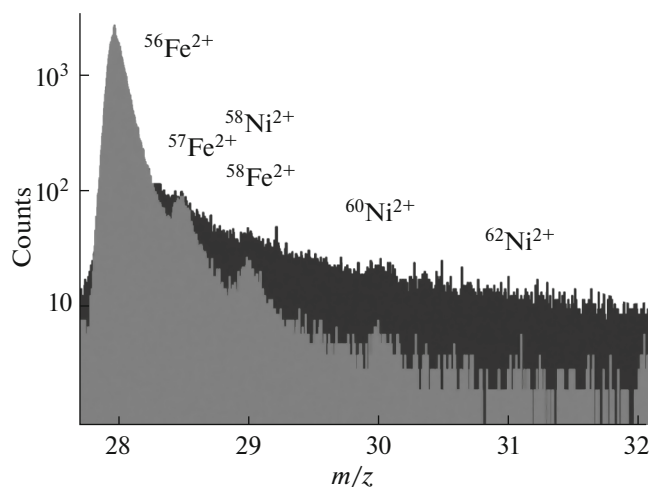


Fig. 3. Mass spectra of Fe-12Cr steel obtained by analyzing specimens with an angle at the tip of $\approx 4^\circ$ (black color) and $\approx 7^\circ$ (gray color).

in the angle at the tip from 4° to 7° , mass resolution $M/\Delta M_{1/10/50\%}$ increased from 7/81/213 to 18/101/219, improving the accuracy of chemical identification (peaks $^{57}\text{Fe}^{2+}$, $^{58}\text{Fe}^{2+}$, and $^{60}\text{Ni}^{2+}$).

Dependence of mass resolution on laser power.

Laser power is an important parameter that determines mass resolution in APT with laser evaporation. It is believed that an increase in laser power with fixed other parameters leads to an increase in mass resolution. This is due to a change in the temperature profile of specimen cooling [12]. To demonstrate this effect, it is necessary to take into account one important factor. As noted above, in performing atom probe tomographic analysis, radius at the tip of the specimen increases and mass resolution correspondingly changes. By simply comparing mass resolution at different laser powers without taking into account the shape of the specimens (for example, changes in its radius), one can obtain contradictory results (see, for example, [13]). For the most rigorous comparison of the data obtained at different laser powers, it is necessary to vary laser power in the experiment so that the shape change is insignificant, or change the power so that the effect of changes in the shape and laser power can be separated. In the present study, a specimen of a Fe-12Cr steel with an initial radius of 40 nm was selected. Thus, in studying this specimen, an increase in radius was accompanied by an increase in mass resolution. If, in the process of such an investigation, there was a transition from a higher power to a lower one, then this transition should lead to the opposite effect: a decrease in mass resolution. Figure 2 shows a characteristic mass spectrum of two consecutive studies of the same specimen, in which laser power was

decreased from 30 to 15 mW. In this case, mass resolution $M/\Delta M_{1/10/50\%}$ decreased from 130 to 107, and $M/\Delta M_{50\%}$, from 175 to 169 with a twofold decrease in power. The observed effect cannot be associated with anything other than a change in laser power, since an increase in radius at the tip during data collection led to the opposite effect. Thus, one of ways to improving the quality of the mass spectrum is to increase the power of the laser that acts on the specimen.

Dependence of the accuracy of the determination of concentrations on laser power. One of the main reasons for the deviation of the detectable chemical composition of the material from the real values in APT is multievents, that is, evaporation of two or more atoms per laser pulse. This is because the detection system is incapable in a number of cases of deciphering such events because of the limitations of time and spatial resolution. As a result, some of the atoms, mostly loosely bound elements, were lost. The selection of APT parameters minimized the number of multievents and increased the accuracy of the determination of concentrations.

Laser power is a parameter affecting the process of field evaporation and, correspondingly, the number of multievents. With increasing laser power, the specimen was heated to higher temperatures, which reduced the difference in the evaporation fields of various elements and, thereby, lowered the probability of the preferential evaporation of elements with the smallest evaporation field [12]. This directly decreased the number of the simultaneous evaporations of individual elements (approximating it to the number of simultaneous evaporations of the remaining elements) not deciphered by the detector, increasing the accuracy of the determination of concentrations.

To assess the accuracy of the determination of concentrations, a model Fe-22Cr alloy was taken, because analogous materials of this class were homogeneous solid solutions of chromium in iron [9], and the average chromium concentrations determined by other methods corresponded to the chromium concentration in the matrix. The chemical analysis of the alloy under study, carried out by X-ray fluorescence spectrometry, yielded chromium concentration of 21.26 ± 0.02 at %. We note that iron has a larger evaporation field than chromium: the evaporation field of Fe atoms is 33 V nm^{-1} , and for Cr, it is 29 V nm^{-1} [4]. Because of this difference in the evaporation fields under nonoptimal conditions of analysis, deviations of the detected concentrations may occur.

The dependence of the detected chromium concentration in the Fe-22Cr alloy is presented in Fig. 5. The operating power range considered was from 1 to 25 mW.

Each point in the graph corresponds to a data set (hereinafter, statistics for the measurement), namely, to 400000 events. The statistical error of the determination of concentration was 0.06%. It can be seen that

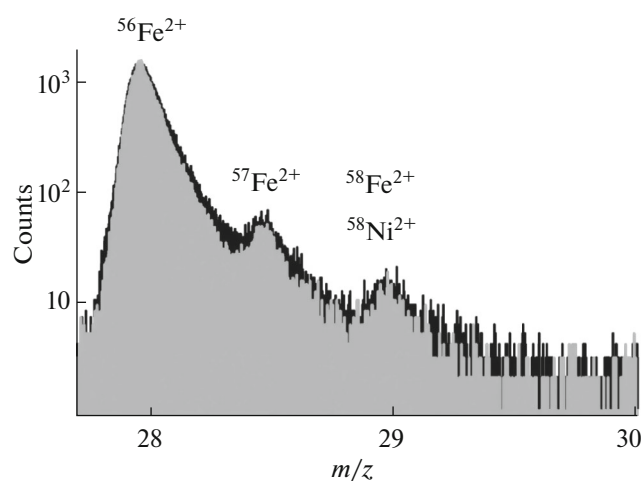


Fig. 4. Mass spectra of Fe-12Cr steel recorded at high power (30 mW, gray color) and low power (15 mW, black color).

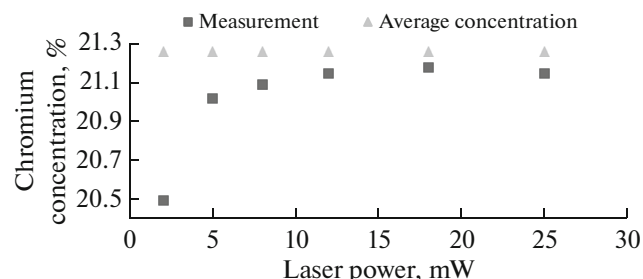


Fig. 5. Dependence of the detected chromium concentration in the Fe-22Cr alloy on the laser power in atom probe tomography.

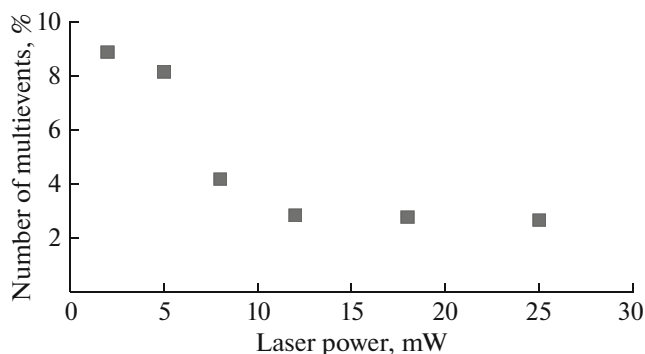


Fig. 6. Dependence of the number of multievents on the laser power when studying the Fe-22Cr alloy by atom probe tomography.

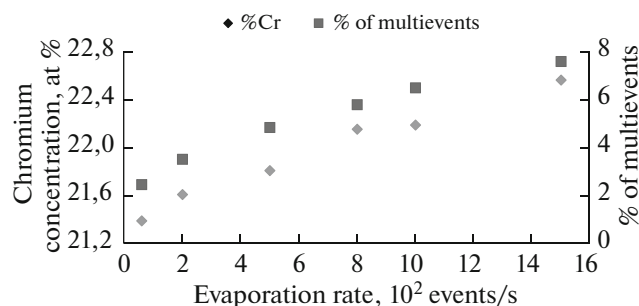


Fig. 7. Dependence of the detected chromium concentration and the number of multievents on the evaporation rate when studying the Fe-22Cr alloy by atom probe tomography.

chromium concentration approached the bulk value as the power increased. In this case, as can be seen from Fig. 6, the number of multievents decreased with increasing laser power.

Thus, with increasing laser power, the accuracy of the determination of concentrations increases. However, it is necessary to take into account the known effects associated with the excessive heating of the specimen: a decrease in spatial resolution [14], a change in the ratio of the degrees of ionization of the evaporated elements, and a change in the composition of the detected ions. Thus, complex molecular ions appear at high laser power, which hinders the interpretation of the mass spectrum and can affect the accuracy of the chemical identification of atoms [13].

Dependence of the accuracy of the determination of concentrations on evaporation rate. One of varied conditions for the APT analysis of materials is evaporation rate (the rate of evaporation of the material). The most characteristic evaporation rate used in atom probe tomographs is approximately 5 events per thousand laser exposures [4], which corresponds to 125 events per second at a frequency of 25 kHz (frequency of actions in the instrument used). This limitation is mainly associated with an increasing error in the determination of concentration because of the growing number of multievents with increasing evaporation rate. To optimize data collection rate, the dependence of the detected chromium concentration in the Fe-22Cr alloy on the evaporation rate was obtained (Fig. 7).

The numbers of multievents for each state are also given in Fig. 7. When collecting data, we used statistics on the measurement of 500 000 events. The statistical error of the determination of concentration was 0.06%.

It can be seen that with increasing evaporation rate, the detectable chromium concentration increased. At the same time, there was a significant increase in the number of multievents, indicating a deterioration in the conditions of an APT experiment. From the point

of view of the accuracy of the determination of concentrations, the conditions with the lowest evaporation rate gave the most reliable data. However, the rate of data collection in this case was the lowest, which may not satisfy the requirement for the amount of data collected for a given time. Therefore, knowing the nature of the dependence of the detected concentration of chromium on an example of the Fe-22Cr alloy on the evaporation rate, one can select the necessary conditions in the analysis of other materials as a compromise between the data collection rate and the accuracy of the determination of concentrations.

CONCLUSIONS

In this paper, we considered the main dependencies of the accuracy of data recovery in the study of chromium steels and Fe-Cr alloys on the variable parameters of the atom probe study using the APPLE-3D setup developed at the Institute for Theoretical and Experimental Physics named by A.I. Alikhanov of National Research Centre "Kurchatov Institute". It is demonstrated that:

- The highest sensitivity of the instrument with respect to elements with a low concentration (by the example of the isotopes $^{60}\text{Ni}^{2+}$ and $^{62}\text{Ni}^{2+}$ in the Fe-12Cr alloy) can be obtained by using specimens with a small initial radius at the tip (approximately 10 nm);

- The mass resolution ($M/\Delta M_{1/10/50\%}$) of the main peak of $^{56}\text{Fe}^{2+}$ increased from 7/81/213 to 18/101/219 with an increase in the angle at the tip from 4° to 7° ;

- The mass resolution at 10% of the height of the main iron peak increases from 107 to 130 with an increase in the laser power from 15 to 30 mW, for example, for the Fe-12Cr alloy;

- The error of the determination of chromium concentration decreases to the level of statistical error with an increase in the laser power from 2 to 25 mW, for example, for the Fe-22Cr alloy;

- The error of the determination of chromium concentration increases to 1% in the Fe-22Cr alloy with an increase in the evaporation rate from 60 to 1500 events/s.

REFERENCES

1. Tolstoguzov, A.B., *Mass-Spektrom.*, 2009, vol. 6, no. 4, p. 280.
2. Rogozhkin, S.V., Aleev, A.A., Lukyanchuk, A.A., Shutov, A.S., Raznitsyn, O.A., and Kirillov, S.E., *Instrum. Exp. Tech.*, 2017, vol. 60, no. 3, p. 428.
3. Kellogg, G.L. and Tsong, T.T., *J. Appl. Phys.*, 1980, vol. 51, no. 2, p. 1184.
4. Gault, B., Moody, M.P., Cairney, J.M., and Ringer, S.P., *Atom Probe Microscopy*, New York: Springer, 2012.

5. Rogozhkin, S.V., Nikitin, A.A., Aleev, A.A., Germanov, A.B., and Zaluzhnyi, A.G., *Inorg. Mater.: Appl. Res.*, 2013, vol. 4, no. 2, p. 112.
6. Aleev, A.A., Iskandarov, N.A., Klimenkov, M., Lindau, R., Moslang, A., Nikitin, A.A., Rogozhkin, S.V., Vladimirov, P., and Zaluzhnyi, A.G., *J. Nucl. Mater.*, 2011, vol. 409, no. 2, p. 65.
7. Rogozhkin, S.V., Ageev, V.S., Aleev, A.A., Zaluzhnyi, A.G., Leont'eva-Smirnova, M.V., and Nikitin, A.A., *Phys. Met. Metallogr.*, 2009, vol. 108, no. 6, p. 579.
8. Rogozhkin, S.V., Bogachev, A. A., Kirillov, D. I., Nikitin, A.A., Orlov, N.N., Aleev, A.A., Zaluzhnyi, A.G., and Kozodaev, M.A., *Phys. Met. Metallogr.*, 2014, vol. 115, no. 12, p. 1259.
9. Rogozhkin, S.V., Korchuganova, O.A., and Aleev, A.A., *Inorg. Mater.: Appl. Res.*, 2016, vol. 7, no. 2, p. 210.
10. Kelly, T.F., Vella, A., Bunton, J.H., Houard, J., Silaeva, E.P., Bogdanowicz, J., and Vandervorst, W., *Curr. Opin. Solid State Mater.*, 2014, vol. 18, no. 2, p. 81.
11. Houard, J., Vella, A., Vurpillot, F., and Deconihout, B., *Phys. Rev. B: Condens. Matter Mater. Phys.*, 2010, vol. 81, nos. 1–5, 125411.
12. Yiyu, Tu., Plotnikov, E.Y., and Seidman, D.N., *Microsc. Microanal.*, 2015, vol. 21, no. 2, p. 480.
13. Prakash Kollia, R. and Meisenkothen, F., *Microsc. Microanal.*, 2014, vol. 20, no. 3, p. 768.
14. Cerezo, A., Clifton, P.H., Gmberg, A., and Smith, G.D.W., *Ultramicroscopy*, 2007, vol. 107, no. 9, p. 720.

Translated by O. Zhukova