

APPLICATION OF COMBINED METHOD TO ESTIMATION OF BRINELL HARDNESS VALUE SPREAD AND DETERMINE THE DEPENDENCE ON THE OXYGEN CONTENT FOR REFINED HAFNIUM

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One of the priority tasks in the operation of existing and creation of new generation nuclear reactors is to ensure safety and increase the resource of regulatory bodies. The solution to this problem can be provided, in particular, through the use of promising structural materials of neutron absorbers. Due to the complexity of its physicochemical and mechanical properties, hafnium is such a material [1].

Today in Ukraine at “Zirconium” State Research and Production Enterprise (Kamyanskoye city), a fundamentally new fluoride calciothermic technology for the production of pure hafnium is developed and mastered. Its peculiarity is that hydrometallurgical redistribution in the production of pure hafnium is part of zirconium purification technology. It is characterized by the identity of operations and is distinguished only by the increase in the number of stages of extraction separation. The process of hydrometallurgical redistribution is completed with the preparation of a hafnium fluoride compound – hafnium tetrafluoride (HfF_4). The calciothermic reduction process of hafnium tetrafluoride with subsequent double electron beam melting of the ingots is at the heart of the metallurgical redistribution. The choice of hafnium tetrafluoride as salt for calciothermic recovery is due to its physical and chemical properties, as well as the possibility of its deep sublimation purification from oxygen (O_2) and nitrogen (N). Calcium (Ca) hafnium tetrafluoride recovery with the addition of 5% of iron (Fe) or aluminum (Al) to activate the chemical reaction of recovery allows to obtain compact ingots of hafnium, and electron-beam melting makes it possible to clear it completely from many impurities. This scheme allows obtaining hafnium with a total content of hafnium and zirconium, which is not less than 99.92%, having a high absorption capacity, and suitable for efficient use in nuclear power engineering.

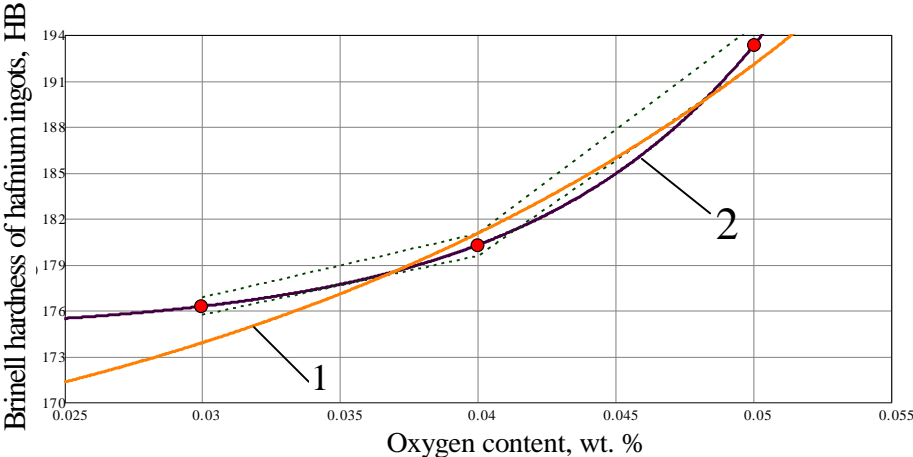
The increase in purity of hafnium leads to the decrease of its hardness after electron-beam melting. The process of electron beam remelting consists of melting the initial ingot in a vacuum and its subsequent crystallization. The base mechanism for refining metals from metal impurities when heated in a vacuum is the evaporation of volatile metal impurities. During the remelting process, oxygen and carbon can be removed due to the formation and removal of volatile metal oxides having a higher vapor pressure than that of the refined metal.

Each of the above methods for producing pure hafnium has its advantages and disadvantages, and the resulting hafnium ingots have differences in chemical composition within the technical requirements established for each method. The content of some impurities is limited only by maximum values. In other words, there are uncertainties in the data themselves, which assess the quality of the alloy ingot. This fact affects the accuracy of determining the dependences of hardness on the content of impurities and, in particular, oxygen.

This uncertainty is present in any process of research and evaluation of experimental data. It contains measurement errors, noise, round-off errors, incomplete information, methodological errors that give rise to uncertainty. It is impossible to overcome the conditions of all these uncertainties. An analysis of numerous scientific papers on the processing of experimental data shows that the interval model can be considered the best model in terms of the correctness of processing such data [2–4].

The application of the interval and standard probabilistic approaches is compared to verify the reliability of the results of an experiment to study the mechanical properties of hafnium samples. The presence of "outliers" in the sample of hafnium ingots hardness values was investigated at fixed weight oxygen content: 0.03, 0.04, and 0.05 wt.% [5]. The situation of measurement error limiting the without reliable information about its distribution and checking the sample for consistency by the methods of interval analysis is considered. The classical statistical tests were used: “ 3σ -rule”, “ 2σ -rule”, Lvovsky’s criterion, “ 3σ -rule” for the median of the sample. The application of these methods shows conflicting results since they all assume that the sample values are subject to a Gaussian distribution. But this sample of measurements is small, and the probabilistic characteristics of the errors are unknown since it is impossible to guarantee compliance with the Gaussian distribution. The correctness of the application of interval analysis numerical methods [6] for processing experimental data under conditions of uncertainty and noisy experimental data is shown.

Determination of the dependence of the hardness of hafnium samples on the weight oxygen content is performed by a combined method. The approximating curves are constructed based from the hardness mean statistical values for each subsample $\bar{x} = (173.857, 181, 192)$ and the central actual values for the truncated subsamples (anomalous values are removed) $x_c = (176.24, 180.24, 193.24)$ obtained by the interval method (Figure).



The dependence of the Brinell hardness of hafnium samples on the weight oxygen content: 1 – approximation by mean values of the sample; 2 – approximation by central actual values

As an approximating curve, we considered a dependence of the form $f(x) = a \cdot e^{bx} + c$. Curve 1 in Figure shows the line $f(x) = 3.622 \cdot e^{43.176x} + 160.628$ when the hardness values were statistical mean values; curve 2 in Figure shows the line $f(x) = 0.052 \cdot e^{117.865x} + 174.462$ when the hardness values were central actual values. The dotted lines show the boundaries of the tube built on the uncertainty intervals $H_1 = [175.68; 176.8]$, $H_2 = [179.52; 180.96]$, $H_3 = [192.0; 194.48]$ obtained by the interval

method on the truncated samples. Curve 1 does not pass through all the uncertainty intervals and, thus, gives a rougher estimate of the dependence of the hardness of hafnium samples on the oxygen content.

The use of a combination of interval analysis methods and classical probabilistic methods of experimental processing provides an alternative flexible tool for obtaining a more accurate and complete analysis of experimental data in the presence of incomplete information, noise, measurement outliers, which is typical when researching the hardness of refined hafnium ingots.

References

1. M.M. Pylypenko. *Hafnium: obtaining, refining, properties*. Kharkiv: FOP Panov A.M., 2020, 236 p.
2. R.E. Moore, R.B. Kearfott, M.J. Cloud. *Introduction to interval analysis*. Society for Industrial and Applied Mathematics, Philadelphia, 2009, 223 p.
3. S.P. Shary. *The finite-dimensional interval analysis*. Novosibirsk, XYZ Publ., 2016, 606 p.
4. O. Yefimov, M. Pylypenko, T. Potanina, et al. *Materials and decision support systems in the nuclear power industry*. LAPLAMBERT Academic Publishing, 2020, 135 p.
5. N.K. Filatova. *Scientific substantiation of the optimal parameters of industrial technology for the production of hafnium ingots for the nuclear industry*: Doctoral thesis. Moscow, 2016, 157 p.
6. S.I. Kumkov. Interval approach to processing noisy experimental data with multiple measurements under conditions of uncertainty // *International Conference "Contemporary Problems of Applied Mathematics and Mechanics: Theory, Experiment, and Practice"*. 2011.