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Characterisation of Porous Tungsten by Microhardness

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Abstract

One of the applications of tungsten is as high current density dispenser cathode in the form of porous tungsten. It is used as a cathode after being impregnated with an electron emissive material so pore distribution in the part is the most important parameter for its function as a uniform and controlled porosity will lead to a better performance.
In this study, application of microhardness as a characterisation method for uniformity of the pore distribution and homogeneity of the structure is introduced. Optical microscopy and SEM is used to relate the results and porous tungsten structure for a better understanding of the method applied.

Keywords: Porous tungsten, microhardness, homogeneity, cathode

Introduction

The performance of a high electron density dispenser cathode in is closely related to the porous structure of the piece since it is impregnated with electron emissive material which should come to the surface every time electrons are emitted as new feeding material.[1] In this connection simply the pores should be of open type and uniformly distributed.[2] They are obtained by powder consolidation methods i.e. pressing and sintering at high temperatures T > 2000. [3]

In the following 80%th and 50%th porous tungsten slugs obtained from the industry are used as test materials for the determination of the change in density / porosity by microhardness (HV) measurements. Optical microscopy and SEM are used to relate results to the microstructure. It is shown that microhardness can be used as a control mechanism for the homogeneity of the porous tungsten structures and the uniformity of the pore distribution in porous tungsten parts.

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Experimental Procedure

Sintered porous tungsten slugs of 6mm diameter each having different densities; 80%th, 50%th impregnated with epoxy resin are used as test materials. LECO – 400 Microhardness Test Machine is used for the measurements. 200gf is used as load since it gave a clear indent although the porosity of the structure was an obstacle to have an accurate measurement. It was made sure that the grain size of the selected samples was smaller than the indent mark so that the hardness values could reflect the particle – pore inter linked surface. The parts are examined in two regions; centre and edge in order to observe the change in density thus porosity in the sample. Coordinates (x,y) are given to the indents and the distance from the centre is calculated using the simple geometrical relationship for the distance between two points; A (x,y) and B (x,y):

$$D = [(x_1 - x_2)^2 - (y_1 - y_2)^2] \qquad \text{(Eq. 1)}$$

Change of hardness with distance from the centre of the cathode is plotted for each sample in order to see the change in density thus porosity along the surface.

The concept of homogeneity index [4] is applied to porous tungsten cathodes to determine the homogeneity of the porous structure using hardness data.

Numerical results are confirmed by the micrographs from optical microscope and SEM.

Results and Discussion

Microhardness test has been done on the following samples having two different densities :

Sample No	Relative Density (%th)
1	80
2	50

Related graphs are plotted; hardness vs distance and %frequency vs hardness range. Homogeneity index concept is applied in order to relate

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hardness measurements to density and porosity distribution of the sample which determine the sample's homogeneity.

A considerable number of hardness values are taken for each sample to avoid false conclusions.

Homegeneity Index

Microhardness measurements are used calculate a homogeneity index (HI) on the basis of a hardness variation factor, V_H where;

$$V_H = (S^2) / (H_m) \qquad (\text{Eq. } 2)$$

$$S^2 = (1 / n -1) \sum (H - H_m)^2 \qquad (\text{Eq. } 3)$$

S: Standard deviation in hardness value
H: Individual hardness value
H_m: Mean hardness value

$$HI = (S / H_m) \times 100 \qquad (\text{Eq. } 4)$$

According to the homogeneity index concept, the smaller the HI value the higher the homogeneity but as we are comparing samples with different densities and since the different is about 30%, HI values are not sufficient to make a conclusion. Frequency of hardness range gives a better view of the structure no matter the difference in densities since it is purely compared on the basis of the distribution of the hardness values. Accordingly, the broader the distribution less the homogeneity. This suggests that, individual hardness values are very much dispersed and the porosity is not evenly distributed similarly density varies along the area of interest so porosity is not uniform and the structure is heterogeneous.

From the figures presented below, it is observed that hardness increases for both of the samples as we go from centre to the edge region. This change in hardness thus porosity is confirmed by the optical photos and SEM micrographs. Samples are first examined individually then are compared with the help of combined plots. The graphs of sample 1 and 2 are given consecutively. As can be seen from the figures, data shows significant scattering. However for the sake of illustrating the trend, linear regression to the obtained data was carried out.

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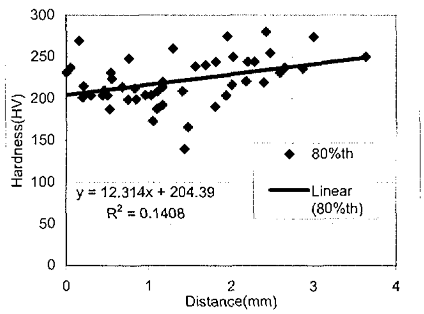

Fig.1.Change of hardness with distance from the centre of the 80%th dense porous tungsten.

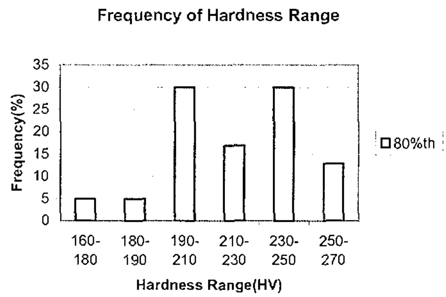

Fig. 2. Frequency of hardness range of 80%th dense porous tungsten.

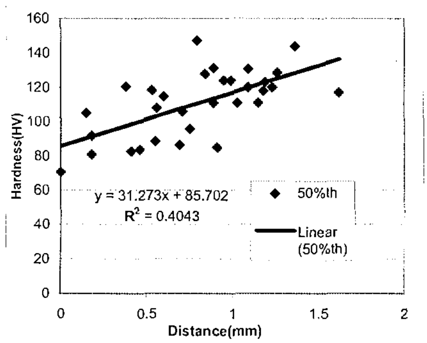

Fig. 3. Change of hardness with distance from the centre of 50%th dense porous tungsten.

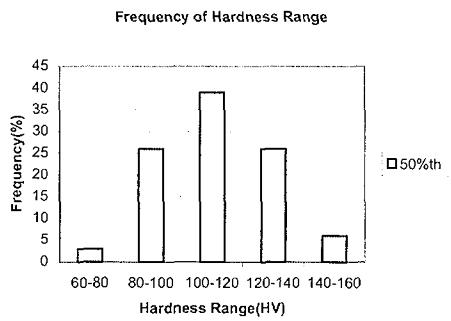

Fig.4. Frequency of hardness range of 50%th dense porous tungsten.

Sample 1 (80%th dense porous tungsten)

The edge region is found to be more porous but still harder than the central region.

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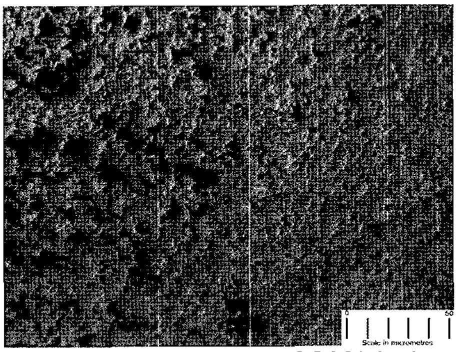

Fig. 5. Optical photo of 80%th dense porous tungsten. (x 20)

Fig.6. SEM micrograph of 80%th dense porous tungsten. (x400)

H_m (edge) = 236.84 HV
H_m (centre) = 204.26 HV

The two regions are compared between each other in terms of hardness gradient and homogeneity. The following figures are related to central and edge regions respectively.

Central Region

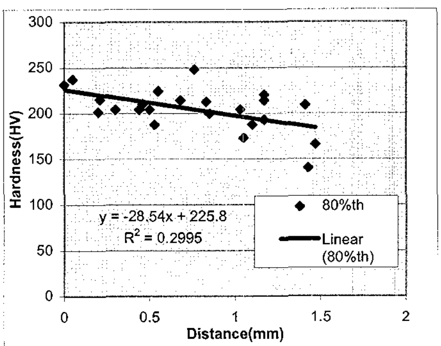

Fig.7.Change of hardness with distance for the central region.

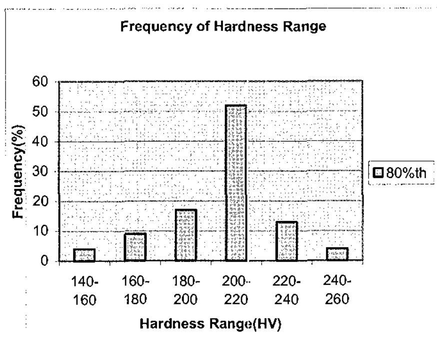

Fig. 8. Frequency of hardness range for the central region.

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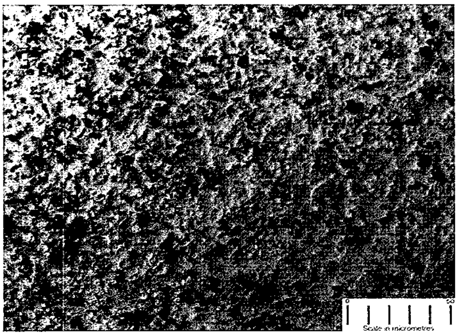

Fig. 9. Optical photo of the central region of 80%th. (x20)

Fig. 10. SEM micrograph of the central region of 80%th. (x1100)

Edge Region

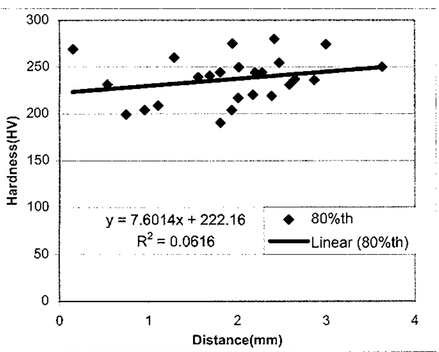

Fig. 11. Change of hardness with distance for the edge region.

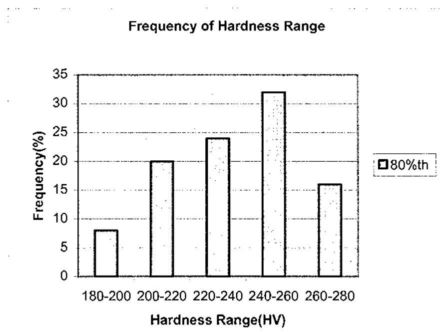

Fig. 12. Frequency of hardness range for the edge region.

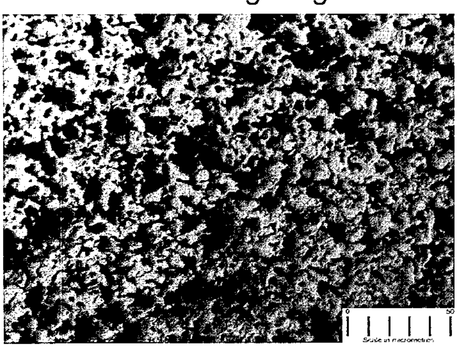

Fig. 13. Optical photo of the edge region of 80%th. (x20)

Fig. 14. SEM micrograph of the edge region of 80%th. (x1100)

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Table 1

Sample No	H_m (HV)	S	V_H	HI
1 (centre)	204.26	23.98	1.68	11.74
1 (edge)	236.84	24.72	1.61	10.44

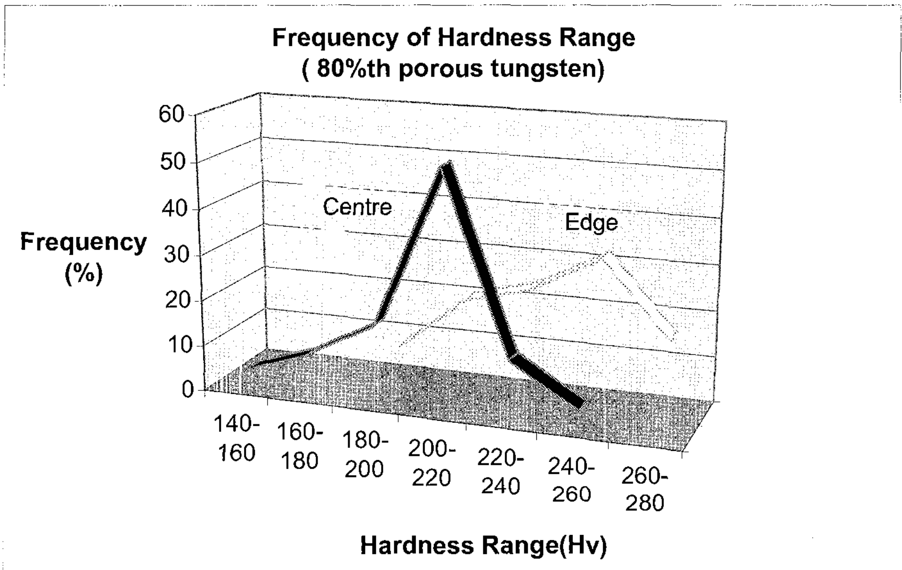

Fig. 15. Comparison of the frequency of hardness ranges of the central and edge regions of the 80%th dense porous tungsten sample.

Sample 2 (50%th dense porous tungsten)

Similarly hardness is observed to be increasing as distance from the centre of the cathode increases along the surface. The increase is observed to be much more than that for sample 1 as the slope of the is higher: $m_2 > m_1$. Central and edge regions are again compared. Optical and SEM micrographs are taken in order to have a better view.

Central Region

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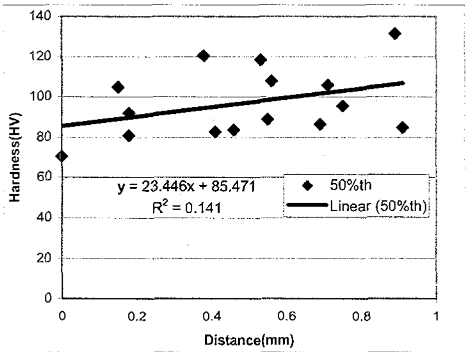

Fig. 16. Change of hardness with for the central region.

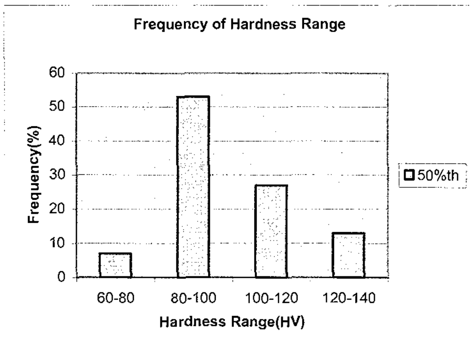

Fig. 17. Frequency of hardness range for the central region.

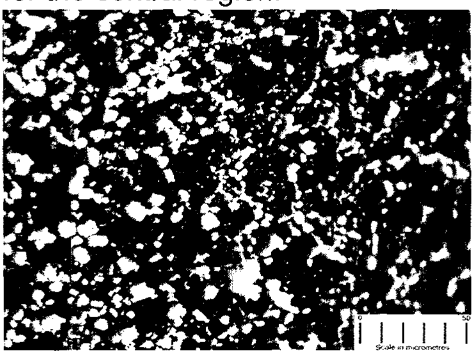

Fig. 18. Optical photo of the central region of 50%th. (x20)

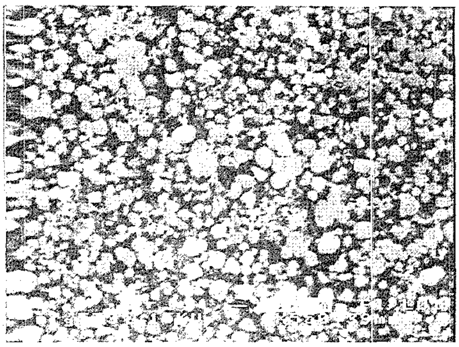

Fig. 19. SEM micrograph of the central region of 50%th. (x550)

Edge Region

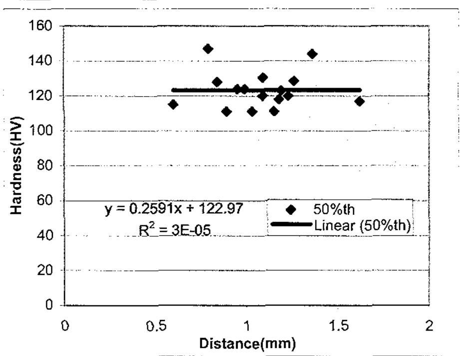

Fig. 20. Change of hardness with distance for the edge region.

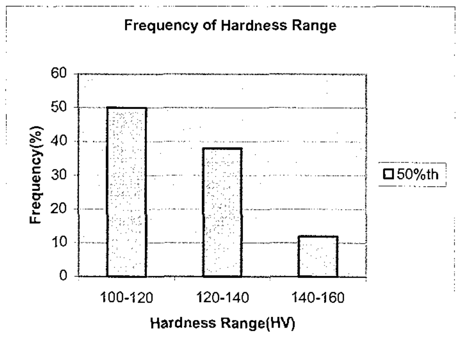

Fig. 21. Frequency of hardness range for the edge region.

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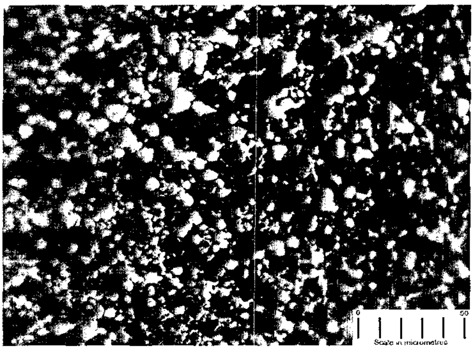

Fig. 22. Optical photo of the edge region of 50%th. (x20)

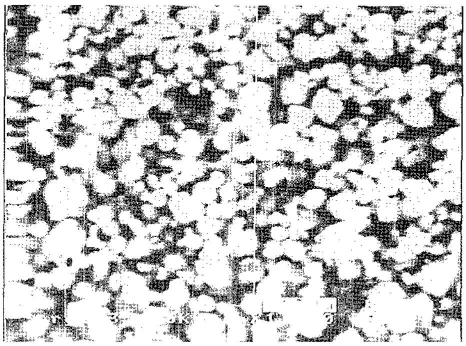

Fig. 23. SEM micrograph of the edge region of 50%th. (x1100)

Table 2.

Sample No	H_m (HV)	S	V_H	HI
2 (centre)	96.96	17.26	1.75	17.80
2 (edge)	123.25	10.66	0.96	8.65

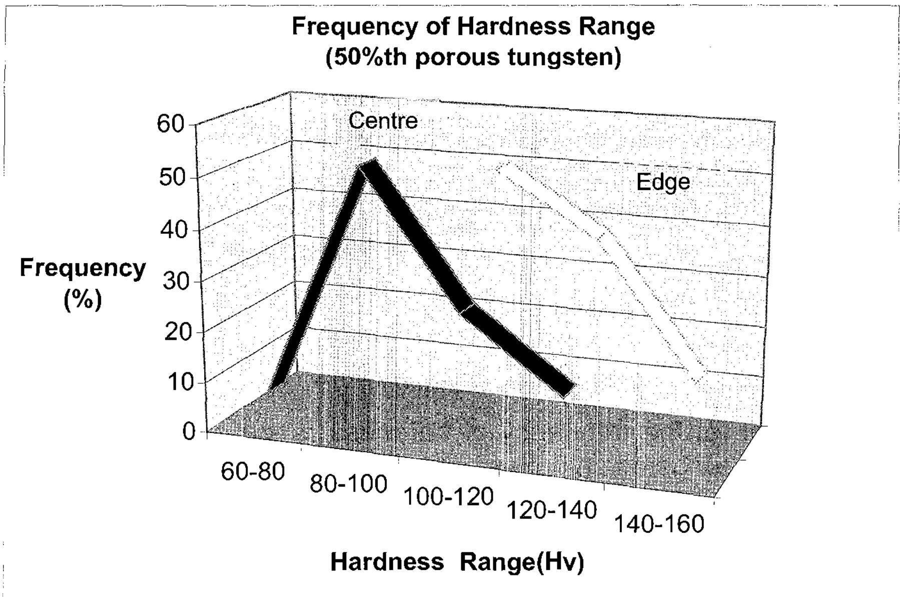

Fig. 24. Comparison of the frequency of hardness ranges of the central and edge regions of the 50%th dense porous tungsten sample.

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Central and edge regions are found to be equally homogeneous with respect to the hardness distribution. Although according to the HI values a relative comparison of homogeneity can be made. Optical and SEM pictures show that the porosity is equally distributed on the surface.

80%th vs 50%th dense porous tungsten cathodes

Finally porous tungsten cathodes with different densities are compared in terms of homogeneity of the structure by plotting the frequency of hardness range for each sample. (Fig. 25)

Combined Plot

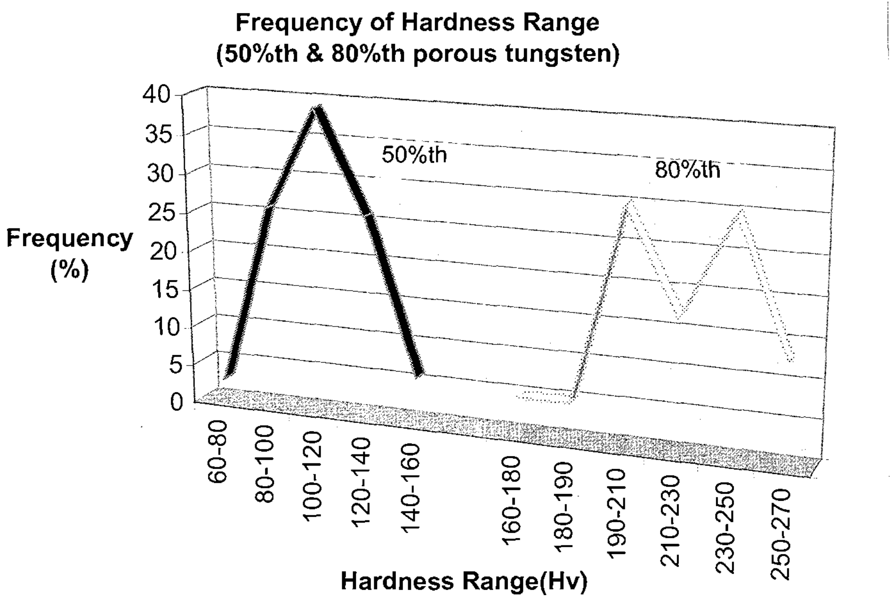

Fig. 25. Comparison of the frequency of hardness range of the 80%th and 50%th dense porous tungsten cathodes.

It is observed that 50%th porous tungsten has a more narrow distribution so it is relatively more homogeneous than 80%th porous tungsten which has actually two distinctive distributions with a discontinuity in the trend, since central and edge regions differ significantly in terms of porosity. The calculated HI values for each sample also confirm the above observation as

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$HI_2 < HI_1$ which means that the 50%th porous tungsten has a more uniformly distributed porous structure than the 80%th dense porous tungsten cathode. Related SEM and optical microscope photos support the results.

On the other hand for both of the samples an increase in hardness from centre to the edge region is observed which can be due to the die wall effect since in the die pressed samples due to the die wall friction particles in the vicinity of die wall are highly packed, deformed and thus hardened resulting in a higher density thus lower porosity and higher hardness as observed. In the 80%th dense sample this effect can be observed more clearly whereas in the 50%th sample since the structure is highly porous it is more difficult to differentiate the centre and edge regions form the micrographs.

As the mean hardness values for each region of the samples are different, HI values change. Since the HI and H_m are inversely proportional, a region with high hardness value will have lower HI which might lead to the conclusion that it is more homogeneous due to the fact that, lower the HI higher the homogeneity. In conclusion, looking at the HI values is not sufficient to compare the samples in terms of homogeneity. Frequency of hardness range should be taken into consideration.

Conclusion

Porosity change of porous tungsten cathodes with different densities is determined by microhardness measurements. Change of hardness along the surface of the sample is examined and related to density / porosity distribution. It is shown that microhardness can be used as a measure for the homogeneity of the porous tungsten structures and the uniformity of the pore distribution in porous tungsten parts.

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