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Influence of grains and grain boundaries on hardness values

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Abstract. Many physical and mechanical properties of materials are connected with their microstructure. Recently, technologies to control the microstructure of materials have been well developed in order to more accurately determine the necessary properties. Therefore, the hardness values of samples from technically pure copper with different average grain sizes are determined. The effect of grain boundaries on the hardness values of a sample from a homogeneous copper structure is studied. The grains effect of different structural components and grain boundaries effect on the hardness values of a sample from steel C45 were also investigated. The grains number in the indentation zone was determined to more correctly obtain the hardness of materials with grains of different structural components, for example, perlite and ferrite.

1. Introduction

Hardness, as a property of the material to resist plastic deformation induced by mechanical indentation, is widely used in materials science [1-3]. It is known that any piece of metal consists of a cluster of many small crystals, which are called grains. The grain boundary surfaces are known as grain boundaries. Hardness has a connection with the structure of materials, for example, with the grain size, the distance between the particles of the intermediate phase, the lengths of slip bands, etc. [4-6]. It was established that a decrease in the grain size leads to an increase in the yield strength. Hall (1951) [7] and Petch (1953) [8] related the measured uniaxial yield stress $\sigma_{0.2}$ of a polycrystalline metal to the grain size d_0 by the relationship:

$$\sigma_{0.2} = \sigma_i + kd_0^{-1/2} \quad (1)$$

where σ_i is the resistance to movement of dislocations in the grain (resistance to friction in the crystal lattice); k is the coefficient characterizing the stress for the activation of dislocations in the grain. The Hall-Petch equation (1) is based on the fact that grain boundaries act as barriers to the dislocations' movement. The influence of grain size on the hardness values of Brinell and Vickers were particularly studied by A.Yu. Marchenkov [9]. He carried out experiments on two materials: steel 10 and pure copper. As a result, it was found that both the hardness at the yield point $HB_{0.2}$ and the Brinell hardness HB increase with decreasing grain size. In work [9] it was also established that the dimensions of the structure elements (average grain diameter, the distance between the particles of the intermediate phase, the length of the slip bands and others) have the same qualitative effect on the hardness of metals and alloys.

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However, the influence of grain boundaries on the hardness of a homogeneous material and a material with grains of various structural components has been little studied. Therefore, in the present work, studies were carried out to investigate the influence of grains and grain boundaries on the hardness of materials.

2. Materials and experimental process

Copper samples with a homogeneous structure and a different average grain sizes and a sample from steel C45 were used. Steel 45 refers to high-quality carbon steels that are widely used in mechanical purposes and industry. At first, the specimens were prepared for analyzing the microstructure and determining the grain size. Samples were pressed into the compound by hot-pressing using the Buehler SimpliMet 1000 machine. Grinding and polishing of the samples were performed on a Buehler EcoMet 250 machine.

To reveal the microstructure, the specimens were additionally subjected to chemical etching. Copper specimens were etched by polishing with a reagent (100 ml H2O + 30 ml HCl + 5 g FeCl3). The specimen of steel C45 was etched in a 4% solution of nitric acid (HNO3) in ethyl alcohol. For the metallographic study, an automated Instron Tukon 2500 hardness tester was used, which allows transferring the image of the resulting indentation to a PC screen with an increase of up to 800 times.

Brinell hardness was determined on a device MEI-T7 using a spherical indenter with different diameters D (D = 0.4, 1 mm) for copper samples and (D = 0.4, 1, 2.5 mm) for a sample of steel C45. The indentation load F was chosen according to standard ASTM E10 (standard Test Method for Brinell Hardness of Metallic Materials) $F=10\ D^2$ for copper, and $F=30D^2$ for steel 45. Vickers hardness was determined using an automated InstronTukon 2500 hardness tester.

3. Results and discussion

The optical microstructure images of copper with different average grain sizes are shown in figure 1. It is clearly that the structure consists only of pure copper. The average grain sizes are 64, 237, 417 microns. Table 1 presents the results of hardness test: indents diameter d and hardness values HB of copper.

Table 1. The results of hardness test: indents diameter d and hardness values HB of copper for indentation by a spherical indenter with different diameters D.

Average grain size, (μм)	D=0.4(mm), F=2(kgf)		D=1(mm), F=10 (kgf)	
	d, (mm)	HB, (kgf/mm^2)	d, (mm)	HB, (kgf/mm^2)
64	0.178	61.34	0.467	55
237	0.186	55.38	0.471	54
417	0.190	53.08	0.479	52.1



a b c

Figure 1. The microstructures of copper with different average grain size: a - 64 μm; b - 237 μm; in - 417 μm.

From table 1 the indentation size effect can be observed, i.e. decreasing the hardness value with increasing the indenter diameter D in the same grain size. The influence of the indention size effect also can be noticed when the grain size decreases at the same load, i.e. the smaller the grain size, the greater the hardness values at the same indentation load F.

To study the effect of grain boundaries on the hardness values of pure copper, indentation tests were carried out under laboratory conditions on an InstronTukon 2500 instrument. A sample with an average grain size of 237 μm was chosen. To obtain an indentation inside the grain, it was necessary to select the load F that provides this requirement. In this case, it was found that such a load F = 0.015kgf. Table 2 presents the Vickers hardness values on the grain boundary and inside the grain.

Table 2. Vickers hardness values on the grain boundary and inside the grain.

HV0.015, (kgf/mm^2)	Inside the grain	On the grain boundary
	75 (68…..75)	75 (68….77)

From table 2 it can be seen that the grain boundary does not affect the results of hardness measurement, i.e. the hardness values at the grain boundary and closer to the boundary 75 kgf /mm^2 almost coincide with the hardness values inside the grain 75 kgf /mm^2. Therefore, when determining the hardness of materials with a homogeneous structure, it is not necessary to focus on the location of indentation and on the number of grains in the indentation zone; in this case, only the influence of the indentation size effect should be taken into account.

Next we will study both the effect of grains and grain boundaries on steel 45. The optical microstructure image of steel 45 in the initial state shows in figure 2. The structure of the steel consists of ferrite and pearlite grains in approximately the same ratio. The average grain size is 40 microns.

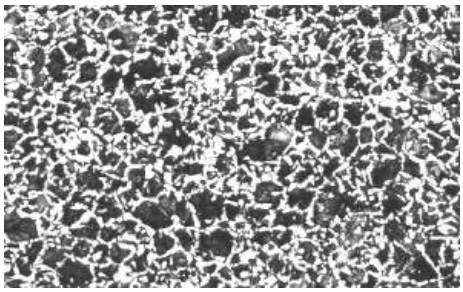


Figure 2. The microstructures of steel 45.

Table 3 shows the results of hardness test: the indents diameter d and hardness values HB of steel 45 by indentation using a spherical indenter with different diameters D. The figure 3 shows the microstructure of steel 45 with indentations obtained by spherical indenters with different diameters D.

Table 3. The results of hardness test: the indents diameter d and hardness values HB of steel 45 for indentation by a spherical indenter with different diameters D.

D=0.4(mm), F=5.26(kgf)		D=1(mm), F=30(kgf)		D=2.5(mm), F=62.5(kgf)	
d, (mm)	HB, (kgf/mm^2)	d, (mm)	HB, (kgf/mm^2)	d, (mm)	HB, (kgf/mm^2)
0.17	220	0.445	182	1.125	179

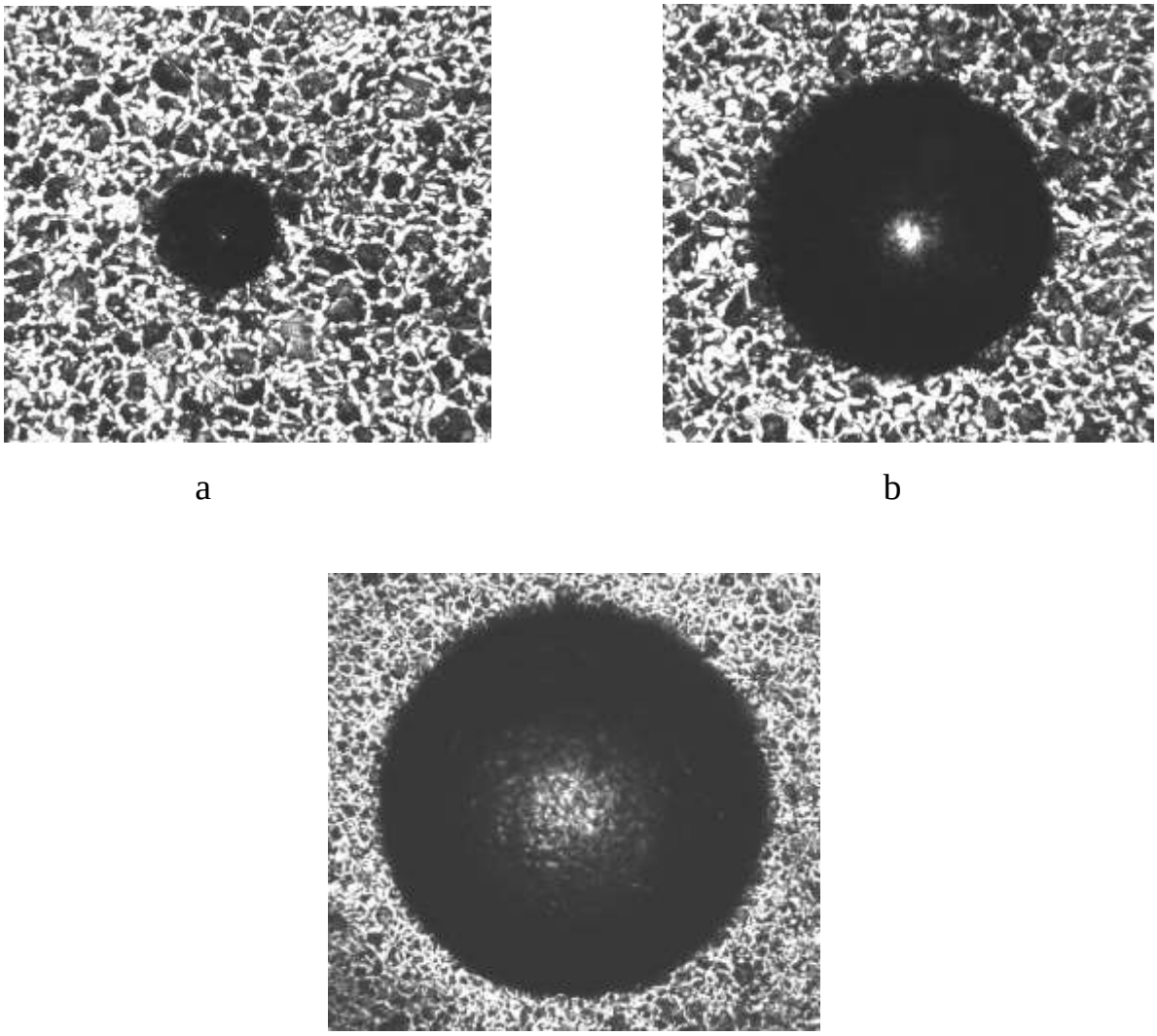


Figure 3. The microstructure of steel 45 with indentations obtained by spherical indenters with different diameters D: a- D = 0.4mm; b- D = 1mm; c-D=2.5mm.

Based on the results presented in the table 3, we can notice the influence of the indentation size effect, i.e. a decrease in the hardness value with an increase in the diameter of the indenter D. Analysis of the results shows that a sufficiently large number of grains (at least 30 in the indentation zone) does not affect the determined values of hardness. This means that, firstly, one should not be afraid of the influence of individual grains of ferrite and perlite on the integral hardness of steel; secondly, it is possible to sufficiently accurately determine the hardness at any indentation load, taking into account the influence of the indentation size effect. For further study of the grain boundaries influence on the hardness values of steel 45 the following experiments were performed.

The main goal of the experiments is to separately determine the grain hardness of the pearlite, ferrite and several grains of pearlite-ferrite, i.e. indentation was carried out at the grain boundary or at the intersection of the grains. Depending on a grain size 40 μm, to obtain an indentation inside the

grain, it was necessary to choose an indentation load F that would achieve this requirement. In this case, it was found that such a load is F = 0.01kgf. The tests were performed on an InstronTukon 2500 automated hardness tester. The Vickers hardness values of the grains perlite, ferrite and at the grain boundaries for steel 45 are presented in table 4.

Table 4. The Vickers hardness values of the grains perlite, ferrite and at the grain boundaries for steel 45.

HV0.01, (kgf/mm^2)	Ferrite grain	Perlite grain	On the boundary
	145 (133…..157)	200 (185….216)	181 (147…..204)

Comparison of the results presented in table 4 with the results of Brinell hardness test in table 3 shows that the hardness value at the grain boundary 170 kgf / mm^2 is close to the value of integral hardness HB determined using an indenter with D = 2.5mm.

In addition, on the same device InstronTukon 2500, the Vickers hardness was determined with an indentation loads 0.1, 1 and 10 kgf; which provided a large number of grains in the indentation. The test results are presented in table 5.

Table 5. The Vickers hardness values of steel 45 with different indentation loads F.

F, (kgf)	HV0.01, (kgf/mm^2)
0.1	180 (160……194)
1	178 (171…..191)
10	177 (172……185)

From table 5 it follows that the Vickers hardness values (from 177 to 180 kgf / mm^2) under different indentation loads turned out to be sufficiently close to hardness at the grain boundary 181 kgf / mm^2, despite the influence of the indentation size effect which decreases the hardness values. Therefore, in this case, for a more justified determination of the hardness value, it is necessary to ensure that the minimum grains number of different structural components in the indentation zone is at least 3-5.

4. Conclusion

The following conclusion can be made by the results of experiments. When determining the hardness of a homogeneous material, the grain boundary does not affect the hardness values determined by different methods with different indentation loads. However, in the case of determining the hardness of materials with grains of different structural components, for example, perlite and ferrite, it is necessary to ensure that the minimum grains number of different structural components in the indentation zone is at least 3-5.

5. References

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