CLASSIFICATION AND STANDARDIZATION OF THE STRUCTURE AND GRAIN SIZE OF BULK METAL MATERIALS

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Abstract
In the 21 century there was an extreme development of nanotechnology, not only in basic and applied research, but especially in manufacturing sector. From these considerations it is necessary to establish precise definitions of basic terms used with regard to applied materials, their structure and size, taking into account their areas of impact, application and use. Sale of products that do not have an incorporated nano-grain and sub-micro-grain structure, nano-particles or are derived therefrom, can not do without standards. The customer in most cases wants a certificate of delivered production on the ground that these are new and unknown facts. The certificate can not be created without standardization of parameters, processes, product use and safety or standards. The further development of research in this area is not possible without standardization of size of grains, which have an impact on increasing the strength and plasticity, change the mechanism of plastic deformation and affect the magnetic properties and the related parameters of materials by ultra-fine, sub-micro-grain or nano-grain structure.

Keywords: standardization, ultra-fine grain steel

1. INTRODUCTION
This article deals only with the question of classification of grain-size of bulk materials. Currently ultra-fine grain and sub-micro fine grain structures are prepared in steel and metal materials, like Ti, Al, Cu, etc. Current standard for determination of grain size, ISO 643:2003, no longer meets these requirements, because it ends with grain size of 17, which corresponds to a mean grain diameter of 1 micron. At the same time it is now possible to obtain mean grain size of 180 micron [1] or 50 micron [2].

It is also proved that refining of the grain size under 1 micron results in changes of parameters and properties of such modified materials compared to structures with medium grain size approximately above 8 micron, grain size number 11.

The grain size in bulk steel and metal materials especially influence their:

- strength and plastic properties
- magnetic properties and related parameters
- mechanism of plastic deformation.

Bulk materials are environmentally safer than the preparation of nanoparticles and nanomaterials made subsequently therefrom.

2. THE GRAIN SIZE IN BULK STEEL MATERIALS
Theoretically, the minimum grain size of steel for nanoparticles can be equal to the size of crystal grid of ferrite or austenite. This consideration does not apply to solid steel materials which are ferromagnetic. This
demonstrates that in these materials there are domains and domain walls. Given that the size of domains is approximately $10^{-5}$ m, the achievement of the medium grain size results in the formation of magnetization after deformation and tensile test of materials prepared in this manner.

For ferromagnetic metals Yensen and Ziegler [3] discovered significant dependence of coercivity of in the mean grains area, which can be generally expressed by a hyperbolic dependence

$$H = \frac{\text{konst}}{Q^{0.5}}$$

where $H$ is the coercivity in [Oe], $Q$ is mean grain area [mm$^2$].

This formula shows clearly the link of the magnetic side of the material with grain size.

3. DOMAIN WALLS

Domain walls arise because the individual domains have completely different spin moments, if the orientation of spins in the transition from one domain to another changed at a great leap on a close border, the border would have a high value of exchange energy. From an energy point of view it is therefore more suitable, if the change in orientation of spins is distributed gradually into individual small leaps in a wide limit.

At balanced state is set its final thickness, which is a compromise between the exchange energy and the magnetic anisotropy energy. Magnetic moments then change their orientation along the helix of Bloch wall or in a straight line of Néel wall. They changed the orientation of 180°, we talk about 180 degrees domain wall, at the change of orientation of 90°, it is a 90° wall.

180° domain walls are about 30-50 nm thick and of about and 90° walls reach thicknesses of about 10-30 nm.

180 ° domain walls can be divided into two groups (see Figure 1):

Bloch wall - rotation of the magnetization occurs in a plane that is parallel to the plane of the domain wall,

Néel wall - rotation of the magnetization vector occurs in a plane that is perpendicular to the plane of the domain wall.

![Fig. 1 - Schematic representation of spin magnetization in (a) Bloch domain wall and (b) Néel domain wall](image)

4. EFFECT OF GRAIN SIZE ON THE MECHANISM OF PLASTIC DEFORMATION

Decreasing mean grain size due to the surfaces size increase results in fundamental change in the course and nature of plastic deformation [1]. If the grain size is over 350 nm, the dominant mechanism of plastic deformation is lattice dislocation slip within the grains. This is caused by lattice dislocations generated during plastic deformation inside the grains of dislocation cells (subgrains).
When the grain size is 350-120 nm, plastic deformation takes place in a similar way as in the grain size above 350 nm, where dislocations are generated within the grains, but their annihilation in dislocation cells (subgrains) decreases and their annihilation increases at the grain boundaries with decreasing grain size.

In grain sizes from 120 to 50 nm the mechanism of plastic deformation occurs mainly through grid slip. Inside the grains there are not important sources of dislocations or their accumulation, grain boundaries serve as sources and dislocation gaps.

If the grain size reaches 50-10 nm, the mechanism of plastic deformation is generated by partial dislocations together with slips on grain boundary and grain rotations.

Grain size decrease occurs together with decrease of the number of low-angle grain boundaries and the amount large-angle grain boundaries. With the grain size below 350 nm there are already practically only large-angle grain boundaries.

5. METHODS FOR DETERMINATION OF GRAIN SIZE

For steel and metal materials processed using submicrotechnology and microtechnology, where the grain size reach between 0.1-5 micron, it is partly possible to use the existing procedures described in the standard ČSN EN ISO 643. However it is also possible to choose other methods, which depend on the ferromagnetic part of the steel, and the fact that the grain size is comparable with the size of domains, then it is possible to use the Hall probe interaction with the surface of the given sample. The following three methods can be used for detection of the grain size:

1. Classic electron microscope

To measure the grain size it is possible to use transmission or scanning electron microscope and the grain size is evaluated using the classic method or evaluation programs. In evaluation of the submicrograins the above methods may show some inaccuracies.

2. Scanning probe microscopy

Scanning probe microscopy, known under the acronym SPM generally indicates microscopy, which is used to obtain information on the structure and its size or other specific properties of the sample [4].

The SPM methods generally use tip with a radius of the order of tens of nanometers. In order for the mutual interaction with the examined surface to occur and to make it possible to monitor the desired properties, it is necessary to bring the tip into contact with the sample, respectively to a distance of several nanometers from the surface, while simultaneously moving the tip (sample) in the plane parallel to the surface. SPM achieves very high resolution properties depending on the thickness of the tip and the accuracy of motion of the sample to the tip. These very fine displacements are realized by piezoelectric elements.

Due to the fact that steel is ferromagnetic, in this case it is possible to apply Magnetic Force Microscope - MFM). This method is intended to measure the forces mediated between the tip and the sample by a magnetic force. The condition is that the tip must be magnetically active.

However, for this microscopy it is possible to use other types of tips that are sensitive to magnetic fields, such as magnetoresistive sensors, hall probes sensors, etc. In the event that the tip is in contact with the sample surface, the tip can be easily affected by the surface topography of the sample than the magnetic forces, therefore the microscope must operate in contact-less mode. It is also necessary to satisfy another condition and that the grain size corresponds to the size of domains.
3. Deformation magnetization microscopy

For ferromagnetic steel, to estimate the size of grains it is possible to use Microscopy Deformation of Magnetization (MDM). This method of estimating the size of grains of steel is based on the following principles:

a) the grain size is less than 10 mm, corresponding to the size of the domains
b) deformation of the steel specimen by a tensile test leads to its magnetizing
c) measuring the coercivity of magnetized sample using Hall probe, etc.
d) calculation of grain size according to the formula

\[ H = \frac{\text{const}}{d} \]

and its correction in dependence on the deformation and tensile test parameters, where \( H \) - Coercivity [Oe], const - constant depending on the steel grade, \( d \) – grain size (dimension) in mm.

For iron, in which Yensen and Ziegler [3] found a significant effect of grain size on the coercivity in the form

\[ H = \frac{0.033}{\sqrt{q}} \]

where \( q \) is the mean grain area [mm\(^2\)] and \( H \) is coercivity [Oe].

The method is fast, inexpensive, its accuracy is comparable with the above methods, but it can be used only for ferromagnetic materials for grain sizes below 8 micron and during tension (pressure) test the sample is magnetized.

6. STRATEGIC VISION FOR EUROPEAN STANDARDS

The above facts within the development of solid steel materials with grain sizes of less than 1 micron for the evaluation of their mechanical and magnetic properties and a description of the mechanism of plastic deformation implies a necessity to modify or amend the standard for the evaluation of the size of the grains. ČSN EN ISO 643:2003 Steel - Microscopic determination of grain size specifies a micrographic method for determining the size of ferritic or austenitic grains in steel. Table C1 of this technical standard clearly shows that the maximum number of grain size defined by the methodology of this International Standard is 17 with the associated mean diameter of grain of 0.001 mm, ie 1 micron. The grain size is related to mechanical and physical properties of the steel, and therefore it would be appropriate to develop a standardized methodology for evaluating grains in the solid (bulk) of steel materials smaller than the 0k001 mm up to the size of 0.00001 mm, ie 100 nanometers or below consequent to dimensions of domain walls, and between 10-50 nm Hall-Petch relationship ceases to apply. Below this threshold, the issue is addressed in the scope of nanotechnology. Amendment (extension) of the standard will meet the needs of submicrotechnology for solid (bulk) metallic materials including steel.

The usability of the proposed standardized method will in particular be in the field of cold forming of materials, ie when drawing, extruding, rolling and the development of new technological processes of decreasing of grain size using submicrotechnology due to improvement of the mechanical and physical properties of solid (bulk) steel and metallic products.

For the evaluation of the mean diameter of grain size in terms of the needs of the above, I suggest grain classification according to Tab. 1.
Tab. 1 - Evaluation of grains of metal materials and steel

<table>
<thead>
<tr>
<th>Grain size numbers</th>
<th>Mean grain size [nm]</th>
<th>Grain title</th>
<th>Grains grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 6-12</td>
<td>44 000-5 500</td>
<td>fine grain</td>
<td>FG</td>
</tr>
<tr>
<td>&lt; 12-17</td>
<td>5 500-1 000</td>
<td>ultra-fine grain</td>
<td>UFG</td>
</tr>
<tr>
<td>&lt; 17-20</td>
<td>1 000-345</td>
<td>submicro grain</td>
<td>SMG</td>
</tr>
<tr>
<td>&lt; 20-23</td>
<td>345-122</td>
<td>fine-submicro grain</td>
<td>FSMG</td>
</tr>
<tr>
<td>&lt; 23-26</td>
<td>122-43</td>
<td>ultra submicro grain</td>
<td>USMG</td>
</tr>
<tr>
<td>&lt; 26-30</td>
<td>43-10,8</td>
<td>nano grain</td>
<td>NG</td>
</tr>
</tbody>
</table>

LITERATURE


