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QUANTITATIVE ESTIMATION OF PARTICLES WITH LARGE SIZE VARIATION

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ABSTRACT

Quantitative evaluation of particles of the size more than 1 μm, inhomogeneously distributed in matrix, is successfully carried on by automatic analysis of images obtained with light microscope (LM - QM). Stereological assessment of smaller particles requires applying of equipment with higher resolution, mainly scanning electron microscope (SEM - QM), which enables more accurate measurements of area fraction A_A.
After common LM - QM assessment, the authors have suggested the SEM - QM measurement, but only in the areas among large, already measured particles. Such LM + SEM - QM method seems to join advantages of both microscopy techniques as well as to avoid their drawbacks. It is of importance that area fractions obtained this way are not additive and following formula should be employed:

$$A_A = (A_A)_{LM} + (A_A)_{SEM} - (A_A)_{LM} \cdot (A_A)_{SEM} \leq 1$$

This scheme has been used to assess carbide phase of new nonledeburitic high-speed steel and to correlate carbide size, shape and arrangement with tool wear resistance. The obtained findings are indicative of significant differences between carbides of new tool alloys and conventional grades of high-speed steels (HSS).

Key words: area fraction, particle size, resolution, quantitative metallography.

INTRODUCTION

Quantitative assessments of large (approx above 1 μm) particles, also inhomogeneously distributed in matrix, were successfully performed with automatic image analysis method based on the light microscopy (Riedl et al., 1987; Fischmeister et al., 1988). It should be pointed out that A_A values so obtained are underevaluated, because by necessity they are restricted to only a fraction of the size bigger than resolution of light microscopes.
Stereological investigations of smaller particles require using of equipment with higher resolution (Egg, 1985), predominantly scanning electron microscopes.
The greater part of available quantitative data on HSS's carbide particles concern investigations based on LM images, moreover, information about the size of the smallest measured particles are stated very seldom. Thus it is reasonable to apply simultaneously

advantages of both light and electron microscopy while estimating area fraction of HSS's carbide phase.

MATERIAL

Specimens were taken from forged non-ledeburitic high-speed steel bar (Tbl. 1) of 25 mm in diameter, and then given annealing (850 °C, 4 h), hardening (1150 °C, 7 min) and tempering (2×540 °C, 1 h). The characteristic feature of these new tool alloys is presence of large primary MC type carbides and secondary carbides of M_6C and $M_{23}C_6$ types of size often smaller than resolution of light microscope (Cwajna et al., 1991).

Table 1. Concentrations of relevant elements of the steel investigated in wt.%

C	Mo	Cr	Ti	Nb	V
1.98	5.25	4.26	3.19	2.10	1.16

DETERMINATION OF AREA FRACTION OF CARBIDE PARTICLES BY JOINED LIGHT AND ELECTRON MICROSCOPY METHOD (LM + SEM - QM)

The core of the LM + SEM - QM method lies at measurement with LM followed by assessment with SEM but exclusively among large, earlier measured particles (Fig. 1).

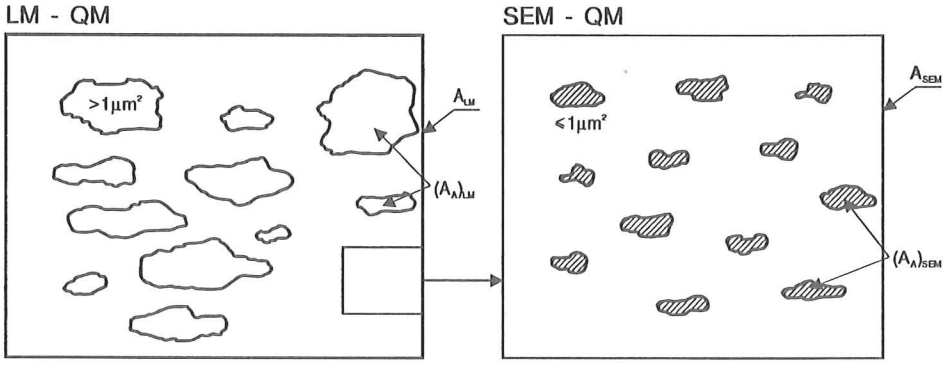

Fig.1. Scheme of A_A assessment by LM + SEM - QM method.

For symbols defined in Fig. 1 the entire area fraction A_A is given by:

$$A_A = \frac{A_{LM} \cdot (A_A)_{LM} + [A_{LM} - A_{LM} (A_A)_{LM}] (A_A)_{SEM}}{A_{LM}} \quad (1)$$

Thus:

$$A_A = (A_A)_{LM} + (A_A)_{SEM} - (A_A)_{LM} \cdot (A_A)_{SEM} \leq 1 \quad (2)$$

which should be observed while performing image analysis according to the proposed scheme.

As a threshold size of particles, 1 μm^2 was accepted; example of structure to be analysed is shown in Fig. 2.

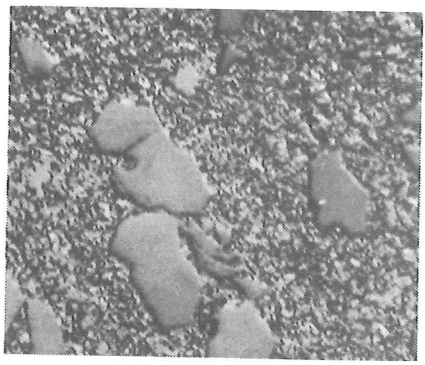

Fig.2. Structure of as-annealed nonledeburitic HSS (LM), polarized light, 775 ×.

It is visible that possibility of automatic detection and consecutive measurements of carbide particles while using LM are restricted solely to large (> 1 μm^2) primary MC type carbides. Therefore, SEM images were employed to assess properly smaller (≤ 1 μm^2) M_6C type carbides in as--annealed and as-hardened steel (undissolved ones) as well as fine dispersion of $M_{23}C_6$ type secondary carbides in as--tempered steel (Fig.3).
All the experimental works were performed with the Morphopericolor automatic image analyser; to improve images quality morphological operations were used.

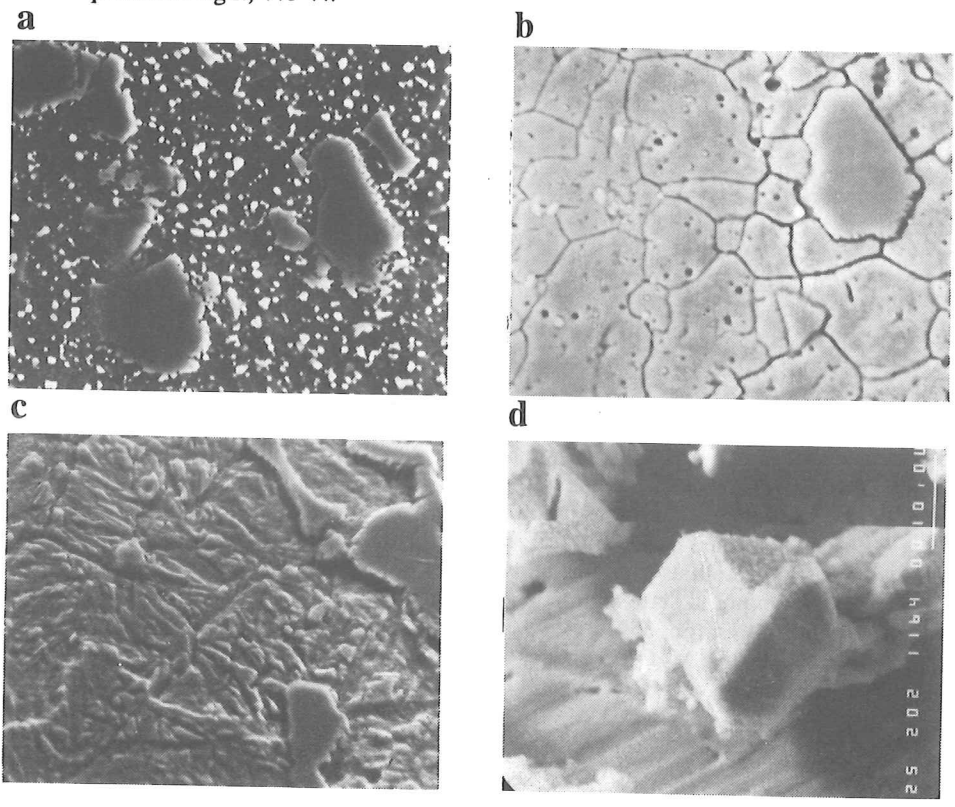

Fig.3. Carbide particles in nonledeburitic HSS (SEM, SEI):
(a) as-annealed state, 1520 ×, (b) as-hardened state, 1520 ×
(c) as-tempered state, 3400 ×, (d) MC type carbide (isolated), 1600 ×

Further step towards entire description of carbides in the investigated tool steel could be based on transmission electron microscope (TEM) images (Fig. 4).

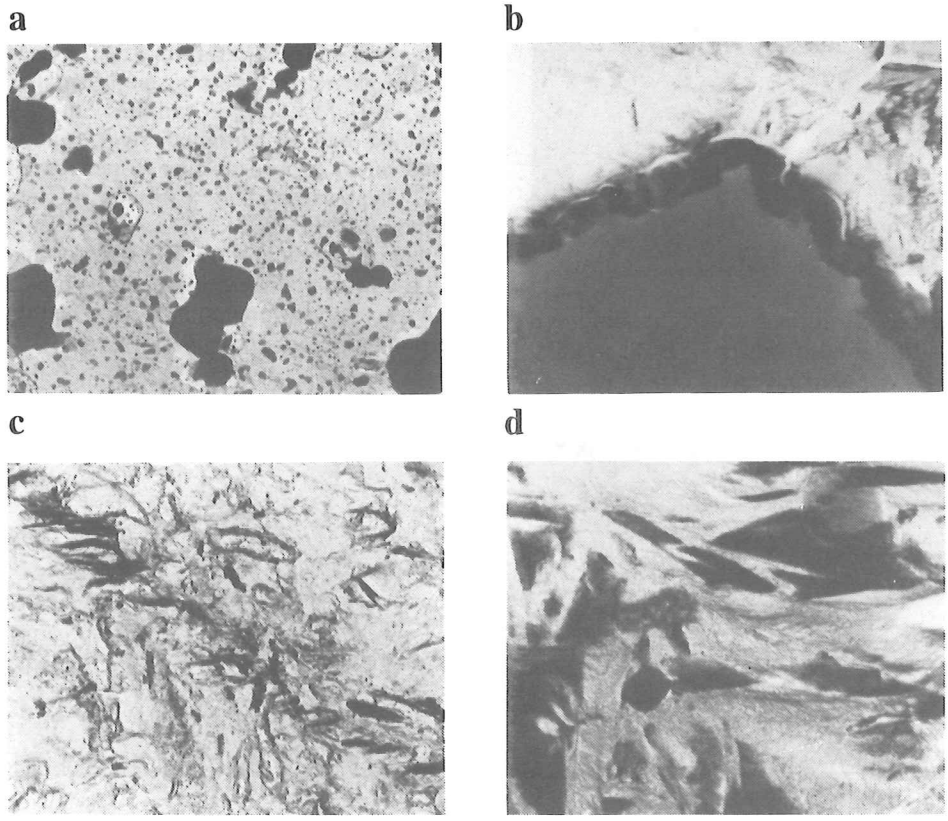

Fig.4. Carbide particles in nonledeburitic HSS (TEM):
(**a**) as-annealed state, extraction replica, 16310 ×,
(**b**) as-hardened state, thin foil, 22230 ×
(M_6C satellites around MC type carbide),
(**c**) as-tempered state, extraction replica, 13000 ×,
(**d**) ditto, thin foil, 35340 ×,
(arrow-like $M_{23}C_6$ secondary carbides and single, undissolved, sferoidical M_6C carbide)

RESULTS

The obtained SEM - QM distributions (both statistical and geometrical) are shown in Fig. 5, mean stereological parameters are collected in Tbl. 2.
Putting the mean values of area fraction into the equation (2) we obtain the following area fractions of carbide particles of the size above 0.05 μm^2: $(A_A)_{annealed} \approx 23.70\%$, $(A_A)_{hardened} \approx 12.77\%$, $(A_A)_{tempered} \approx 18.04\%$.

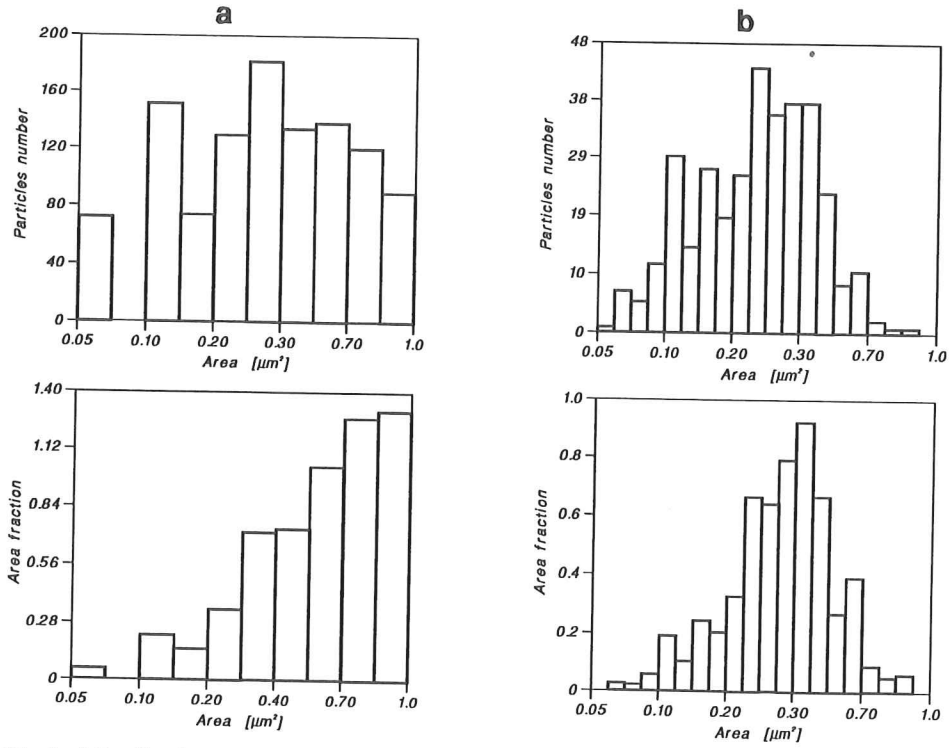

Fig.5. Distribution of carbide particles size:
(a) - M_6C type carbides in as-annealed steel,
(b) - $M_{23}C_6$ type carbides in as-tempered steel

Table 2. Stereological parameters of carbide phase in nonledeburitic HSS.

Measured phase[1]	Microscopic techniques	Shape factor ξ[2]	A_A [%]	$\bar{A}$ [μm^2]
MC, M_6C (sporadically) type carbides, > 1 μm^2	LM	0.67	14.36[3] 12.18[4] 12.49[5]	30.50[3] 28.82[4] 24.84[5]
	SEM	0.48	-	-
M_6C type carbides in annealed steel, ≤ 1 μm^2	SEM	0.69	10.90	0.43
M_6C type carbides in hardened steel (undissolved), ≤ 1 μm^2	SEM	0.71	0.67	0.40
$M_{23}C_6$, M_6C (sporadically) type carbides in tempered steel, ≤ 1 μm^2	SEM	0.40	6.34	0.26

[1] Identified by electron and X-ray diffraction,
[2] $\xi = 4\pi \cdot \text{area}/(\text{perimeter})^2$,
[3] in annealed steel, [4] in hardened steel, [5] in tempered steel.

CONCLUDING REMARKS

In conclusion we can state that:

- the LM + SEM - QM method of dispersed particles of bimodal size distribution area fraction assessment was found to be useful in investigation on carbide phase in new nonledeburitic tool alloys,
- joined LM + SEM method permitted to understand more precisely the phenomena (dissolution and precipitation) during heat treatment of nonledeburitic HSS as well as compare carbide phase of these new steels with that of traditional grades,
- according to expectations (Jost et al., 1988) shape factor of primary MC carbides amounting 0.67, when measured with LM decreased to 0.48, when measured with SEM,
- SEM and especially TEM investigations enabled to establish factors (besides high hardness of TiC and NbC carbides) of high cutting performance of the new steel: regular (octahedral) shape of MC type large primary carbides and their sharp edges, strong embedding of these particles in a metallic matrix due to closely surrounding fine M_6C type carbides, advantageous (arrow-like) shape of secondary $M_{23}C_6$ type carbides. The findings concerning relationships between tool life and steel microstructure testify a necessity of applying microscopes with proper resolution, including the largest available one,
- it was confirmed (Maliński et al., 1989) that to characterize particles size thoroughly it is indispensable to apply both statistical and geometrical distributions,
- quantitative data concerning particles size and area fraction should be supplemented by information about size lower limit of measured objects (connected with the resolution of used microscope),
- further investigations should be directed towards measurement employing higher resolution and magnification i.e transmission microscopy.

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