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QUANTITATIVE STRUCTURAL CRITERIA OF STEEL QUALITY CONTROL

Jan CWAJNA

Silesian University of Technology
Department of Materials Science
Krasińskiego 8, PL-40-019, KATOWICE,POLAND

ABSTRACT

Fundamental features of steel microstructure as well as stereological and morphological parameters applied in their description are presented. A critical analysis of semiquantitative and quantitative criteria for steel quality control is done.
A special attention is paid to fundamental problems of quantitative metallography, i.e. evaluation of:

- content, size and shape of nonmetallic inclusions and other particles,
- volume fraction of phases,
- grain size and shape,
- nonhomogeneity of phase distribution,
- microstructural anisotropy.

The required properties of quantitative structural criteria for steel quality assessment are discussed. Factors affecting intra- and interindividual intrinsic variability as well as the extrinsic variability of the results of quantitative evaluation of steel microstructure are analysed.
It is shown that only a small part of stereological knowledge has found a common application in quantitative description of steel microstructure in materials science and engineering (MSE) studies and particularly in quality control.
Some recommendations, based on the latest achievements in stereology and image analysis, to improve this state are proposed.

Key words: quantitative metallography, steel quality control.

INTRODUCTION

The majority of worldwide used steel grades and methods for their processing are in general a result of many years of practical experiences and tests rather than a result of systematic scientific researches. These last mentioned, served predominantly as confirmation of theoretical principles of earlier accepted chemical compositions and technologies. Consequently, numerous qualitative relations between chemical composition, production process, microstructure and properties have been established. It is highly insufficient for development of new steels, with the a priori assumed microstructure and properties, designed for particular application and service conditions (Maciejny, 1986 and Farge, 1989).
It can be stated that small accuracy and "human sensitivity" of widely used comparative methods of microstructure analysis as well as inadequacy of applied quantitative measures cause a barrier for developing quantitative structure-property relationships and, consequently, for steel development.

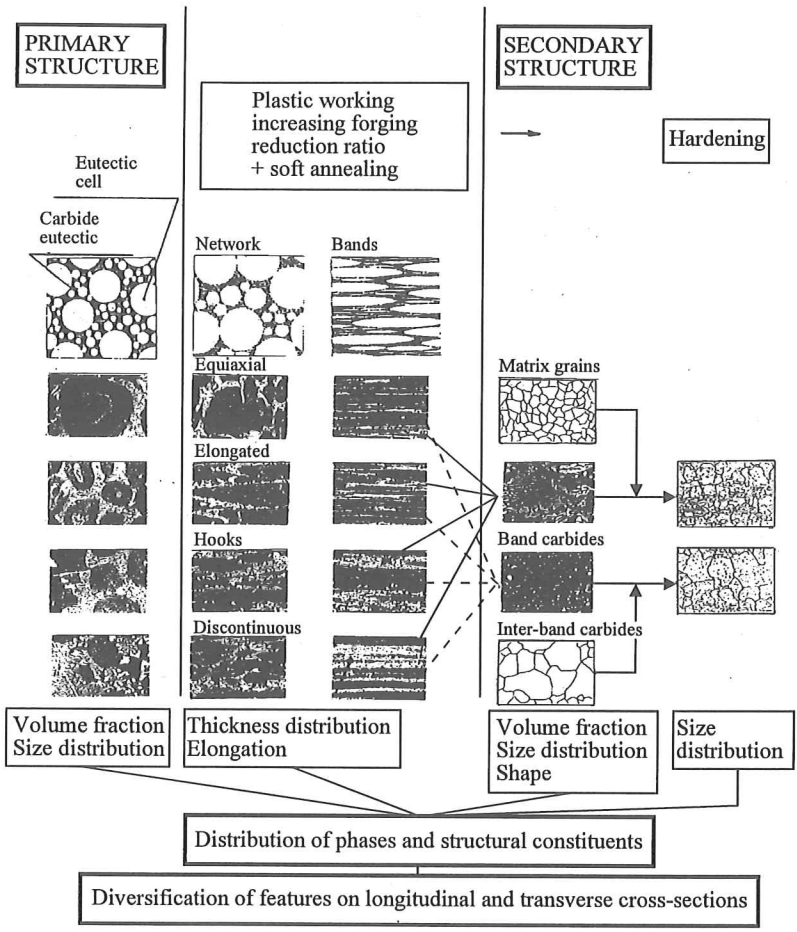


Fig. 1. Model primary structures of high speed steels and corresponding secondary structures in bars with small and large forging reduction ratio.

Significant improvement in classical steels (like high-speed steel) (Riedl et al., 1987) and invention of new kinds of steels, like dual phase (Lanzillotto and Pickering, 1982), duplex (Nicodemi and Roberti, 1992) or nonledeburitic high-speed steels (Cwajna et al., 1991, 1992) was possible due to the application of precise, structure-based criteria of quality assessment while optimizing their chemical composition and production parameters. Simultaneously, for these groups of materials the most advanced theory of properties has been evaluated. In other words, there exists a clear feedback between the steel properties and the complexity as well as precision of quantitative description of its microstructure.

In up-to-date quality control systems the control criteria enabling early elimination of defective semi--finished or final products are of the highest importance. For this purpose structural criteria can be successfully applied (Mc Call and French, 1979).

To conclude, improvement of quantitative criteria for quality assessment of primary and secondary structure of steel is one of the fundamental conditions for further progress in steel production. Moreover, it should enable to reach the higher design level in which the trial-and-error method will be substituted by scientifically based and property oriented methods for development of new materials.

BASIC PARAMETERS FOR QUANTITATIVE ASSESSMENT OF STEEL MICROSTRUCTURE

Contemporary metallography allows to describe the relation between primary and secondary microstructure as well as enables appropriate choice of structural factors affecting technological and mechanical properties of materials or durability and reliability of machine parts made of these materials (see Table 1 and 2, Fig. 1). The above mentioned parameters are:

- volume fraction, number, shape, size and spatial distribution of non-metallic inclusions and dispersed phase particles,
- volume fraction of phases and structural constituents in two- or multiphase steels,
- grain size and shape in granular structures,
- nonhomogeneity of phase distribution,
- anisotropy of structure.

Parameters applied to characterizing structural features are presented in Tables 3 and 4 which were prepared on the grounds of monographs (De Hoff and Rhines, 1968; Underwood, 1970; Saltykov, 1977; Ryś, 1982; Elias and Hyde, 1983; Coster and Chermant, 1985; Exner and Hougardy, 1986; Russ, 1988) and review works (Underwood, 1979, 1987; Rhines, 1985 and 1986; Ryś, 1986; Liu, 1992; Cwajna et al., 1980 and 1981, 1993a, 1993b and Cwajna, 1991).

These sets of parameters demonstrate that nowadays it is already known how to describe the steel microstructure in a quantitative way. The point is in proper choice of stereological parameters and in the measurement methodology which should give results of sufficient accuracy and repeatability while testing industrial products, not only small laboratory specimens. Tables 3 and 4 also testify that stereological knowledge is insufficiently applied in MSE and hardly used in quality control. Thus it seems to be indispensable to explain it's reasons as well as to propose ways of wider spreading the achievements of stereology.

REQUIRED PROPERTIES OF STRUCTURAL CRITERIA FOR STEEL QUALITY ASSESSMENT

An attempt to classify the required properties of structural criteria for materials, taking into account experience in MSE (Coster and Chermant, 1985; Rhines, 1985 and 1986; Coster et al., 1987; Exner, 1987; Chermant and Coster, 1990; Ralph, 1990) as well as in biology and medicine (Kališnik, 1988), is presented in Table 5.

All the available sets of quantitative structural criteria fulfil only in part the requirements listed in Table 5. However, in numerous publications there are clearly pointed the elements of research methodology to be improved in order to achieve satisfactory structure characteristics. In general one should use quantitative instead of qualitative methods and control all factors affecting the results of quantitative methods (Table 6).

The most critical factor affecting the final results of steel microstructure analysis is the sampling strategy. Number and manner of specimen preparation should be chosen accordingly to the repeatability of chemical composition, microstructure and properties within the production method and particular melts. Inhomogeneity in chemical composition and structure (in melts, semi-finished and final products) should also be taken into account.

Any systematic research in this field is very expensive due to high cost of both the material for tests and experiments. Thus, such a complex research can be done only occasionally. It forces very detailed control of all the factors affecting this kind of variation in every research done to establish technology-structure-properties relationship.

Intrinsic variability of the material tested affects the way in which specimens are cut and the number of analysed fields within each specimen. The above mentioned items are fixed on the basis of macro- and microscopical metallographic observations.

Specimen preparation should enable detection of characteristic microstructure features in the whole object volume (Fig.2). So, the first condition for proper quantitative analysis is perfect conventional metallographic inspection.

Research strategy for quality control described in standards (e.g. ASTM E 45) assumes preparation of polished section from the head and bottom of the first, medial and last ingot from the same melt

Table 1. Classification of one-phase steel microstructures - structural features analysed quantitatively.

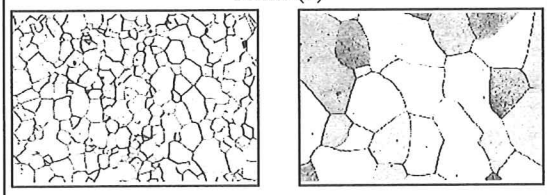
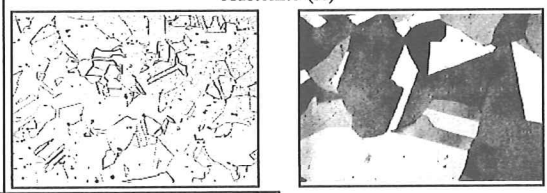
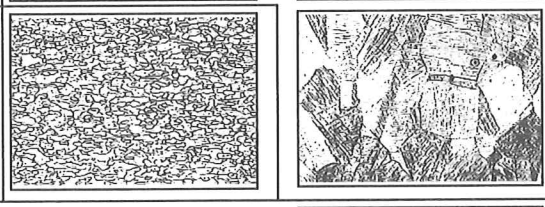
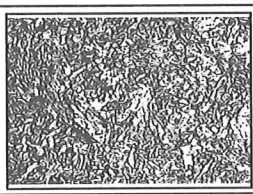
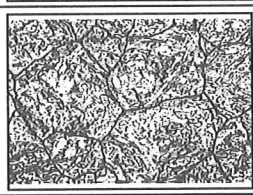
Microstructure		Structural composition	Structural features analysed quantitatively				
			Grain size distribution	Grain shape	Degree of orientation of grain boundaries	Specyfic area of twins	Needle size
Granular	Equiaxial grains	Ferrite (F)  Austenite (A) 	●	●		●	
	Elongated grains		●	●	●		
Acicular		Martensite (M) 					●
Mixed		Martensite with prior austenite grain boundaries 	●	●			●

Table 2. Classification of basic microstructures of two- and multiphase steels - microstructural features quantitatively analysed.

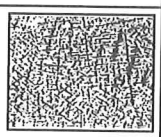
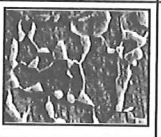
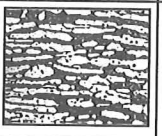
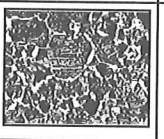
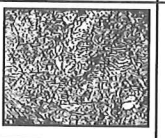
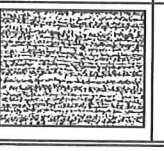
Microstructure				Structural composition Examples	Structural features analysed quantitatively				
					Volume fraction of phases	Number of grains (particles)	Grain (particle) size distribution	Grain (particles) shape	Distribution inhomogeneity of phases
Primary structure	Dendritic				●		□	●	○
	Globular				●	○	●	○	○
	Striped			Ferrite (F) + Perlite (P) 	●		●	●	●
Secondary structure	Granular	Equiaxial grains	Dual phase	(F) + Martensite (M) 	●	●	●	●	●
			Duplex	(F) + Austenite (A) 	●	●	●	●	●
			Discon-tinuous net	(F) + (P) 	●		●		●
			Net	(P) + Carbides (C) 	●	●	●		●
		Striped		(F) + (P) 	●		●	●	●

Table 2. - continuation.

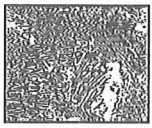
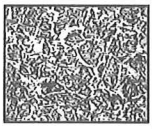
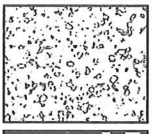
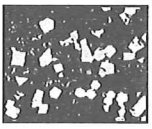
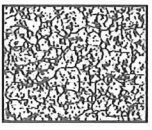
Microstructure			Structural composition Examples	Structural features analysed quantitatively				
				Volume fraction of phases	Number of grains (particles)	Grain (particle) size distribution	Grain (particles) shape	Distribution inhomogeneity of phases
Secondary structure		Lamellar	(P) 	○		□	○	
	Acicular	Widmanstätten	(F) + (P) 	●		□	●	
		Bainitic	(F) + (C) 				●	
	With dispersed particles	Homogeneous	(F) + (C)  (M) + (C) 	●	●	●	●	●
		Net, elongated net, discontinuous net, bands, stringers, clusters	As in Fig. 1	●	○	●	●	●
	Mixed	Granular with dispersed particles	(M) + (C) 	●	●	●	●	●
		Lamellar--Netlike--Acicular	(P) + (C) 	●	○	□	●	●

Table 3. Comparison of parameters use for nonmetallic inclusions assesment in stereology, MSE and industrial quality control.

Features to evaluate	Stereology		MSE		Industrial quality control	
	[1])	Comments and examples	[1])	Comments and examples	[1])	Comments and examples
Contents - phase composition	+	Volume fraction of all and various kinds of non-metallic inclusions (NMI) (selective evaluation: selective and potentiostatic eaching, evaporated layers + dark field, polarized light + special technique of SEM + image analysis) $V_V=A_A=L_L=P_P$			+	- visual classification of inclusions - comparative methods commonly applied (standard charts) - $V_V=P_P$ manual method JIS G 0555-1956 (Japan)[2]), ASTM E 562-86 (USA), BN-77/4054-01 (Poland) automatic methods: ASTM E 1245-89 and ASTM E 1222-86 - evaluation of precision and bias
	++	Well elaborated manual and automatic measurements, precise control and estimation of errors				
Number	+	- N_V, N_A, N_L - well elaborated parameters, week point - N_V for particles of irregular shape - limited possibilities of serial sectioning applications, but possibilies from probabilitic modelling	+	N_L, N_A commonly evaluated; rare cases of N_V estimation	-	- comparative methods commonly applied - N_L, N_A → ASTM E 1245-88
Size	+	- parameters of NMI volume distribution: $\overline{V}$, s(V) - specific surface of NMI: S_V - relative specific surface of various NMI: S_R	o	- comparative methods still often applied - commonly used measures of particles size in 2-D space: L_A, $\overline{A}$, $\overline{l}$ - single examples of 3-D parameters determination	-	- comparative methods commonly applied {ISO 4967; ASTM E 45-87; DIN 50602} - classification on L_A and $\overline{l}$ basis (inclusion rating number) → ASTM E 1122-86 - measurements of $\overline{A}$ and $\overline{l}$ → ASTM E 1245-89
Shape	-	- no versatile parameter - recommended distribution of 2-D shape factors (as a combination of NMI area, perimeter, ferret diameter and moments of interia)[3)]	-	- inclusion malleability factor - distribution of various shape factors	--	only the comparative methods (standard charts)
Distribution	o	- mean free path $\lambda=1-V_V/N_L$ - anisotropy index $AI=N_{L\perp}/N_{L\parallel}$ - degree of orientation $\Omega_{1,2}=N_{L\perp}-N_{L\parallel}/N_{L\perp}+0.571N_{L\parallel}$ - measurements of NMI stringers length and clusters size with automatic image analysers - other methods of not completly verified versatility			o	→ ASTM E 1245-89 and ASTM E 1268-88 → → } ASTM E 1268-88 → - comparative methods commonly applied

Notes: [1]) Denotes the current level of accuracy, adequacy and repeatability of parameters currently in use: ++ very satisfactory, + satisfactory, o medium, - poor, -- very poor, [2]) Examples of standard practice based solely on stereological principles, [3)] but a shape parameter in 2-D has no meaning

Table 4. Comparison of parameters use for steels microstructure assessment in stereology, MSE and industrial quality control.

Features to evaluate	Stereology		MSE		Industrial quality control	
	1)	Comments and examples	1)	Comments and examples	1)	Comments and examples
Contents - phase composition	++	As in Table 3			+	As in Table 3
Number of Grains (Particles)	+	• As in Table 3 • Grain number - empirical equation determined by computer simulation $N_V = \left(\frac{2.415}{\sqrt{N_A}} - \frac{1.4552}{N_L} \right)^{-3} [mm^{-3}]$			o	• Comparative methods (standard charts) {ASTM E 112-88; DIN 50601} • Manual quantitative methods: N_A, N_L [ASTM E 112-88] 2) • Automatic methods: [ASTM E 181-87, ASTM E 930-83]
Grain (Particle) size	+	• As in Table 3 • Mean volume weighted grain volume and coefficient of variation of grain volume: • Jensen, Sorensen method $\overline{V}_{(V)} = \frac{\pi}{3}(\bar{l})^3 [mm^{-3}]$ and $\vartheta(V)_{(V)} = \sqrt{\frac{36k(\bar{A})^3}{\pi[(\bar{l})^3]^2} - 1}$ $k = 0{,}071 \div 0{,}083$ • Mean grain volume: • Empirical equations determined by computer simulation $\overline{V} = 1.3\left(8\sqrt{6}\frac{\bar{l}^3}{\pi^2}\right) [mm^3]$			o	• As in Table 3 • Grain size chart number {ASTM E 112-88} • Manual and automatic methods of determination of $\bar{A}$ and $\bar{l}$ as well as intercept lenght distribution: [ASTM E 112-88; ASTM E 930-83; ASTM E 1181-87]
Shape	o	As in Table 3	-	• Mean shape factors; Rare cases of shape factor distributions application (comment as in table 3)	--	As in Table 3
Distribution	o	As in Table 3				

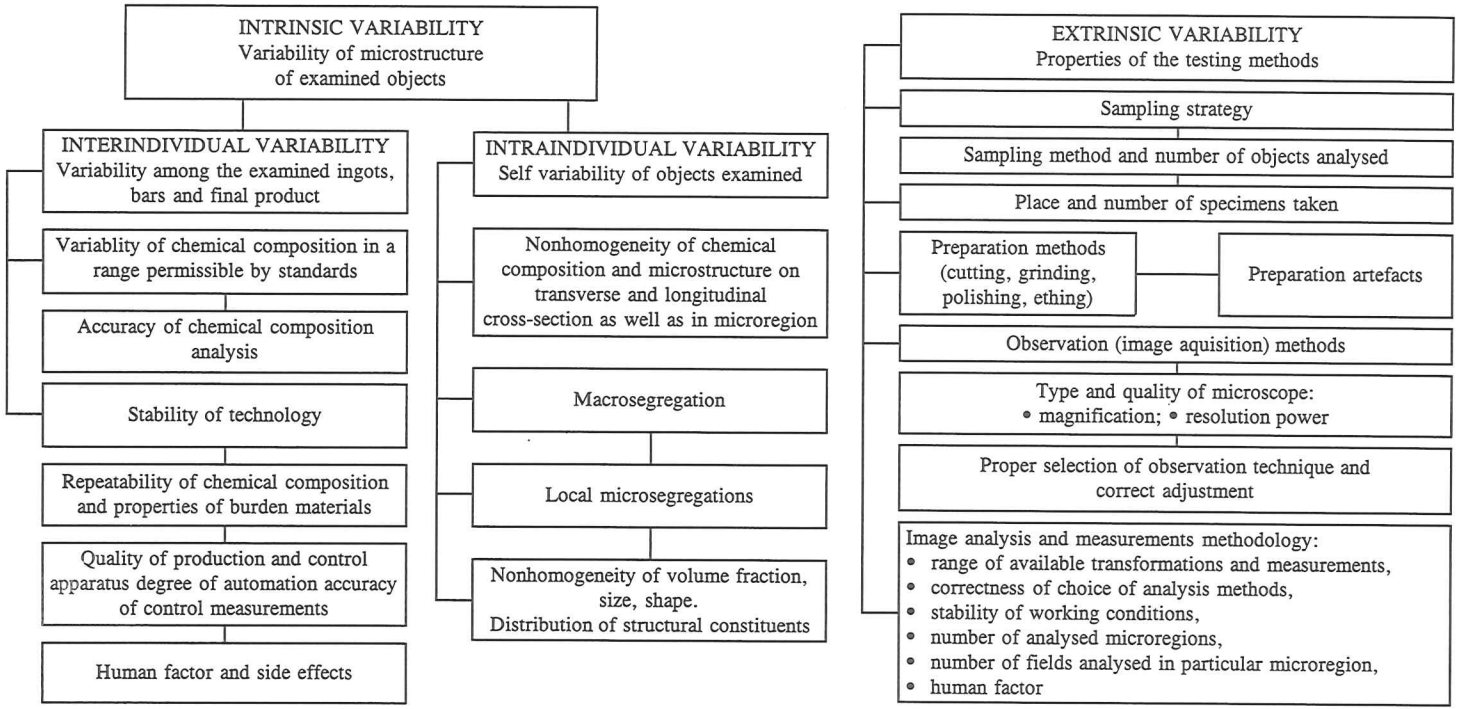
1) As in Table 3

after plastic working. Occasionally specimens are cut from the inner part of the ingot. Usually the specimens are taken from the mid-thickness or at the quarter-thickness location. Structural maps in Fig.2 show that such strategy in not quite perfect. This conclusion is also supported by results of non-metallic inclusions contents tests performed by McCall and French (1979). Accordingly to their results satisfactory accuracy of this measure can be obtained after examining at least 100 specimens taken from each melt. This aspect should be interpreted in further works on quantitative structure evaluation.

Table 5. Required properties of structural criteria for steel quality assessment.

Validity		Univocal relations with properties of the material or final product
Versatility		Possibility to compare structural constituents of various steels localized at any place in the material volume
Objectivity		Perfect description of a model (master) structure
Invariance by translation or rotation		Morphological information obtained on a structure must be independent of the position of frame of measurements
Homogeneity		If measurements are done at several magnifications on the same set, the results must be the same
Continuity		A small deformation of the structure must not lead to large changes in the parameters measured
Additivity		$W(X) + W(Y) = W(X \cup Y) + W(X \cap Y)$ were **W(X)** is the measure of parameter **W** on set **X**
Accuracy	Unbiased sampling design	Representativity - a sufficient number of sample units in order to keep relative error reasonably small and to take the representative sample - all the units of a population have the same chance of being selected for the sample
	Precision	Satisfactory precision usually is obtained when the standard relative error is less or equal to 5%
	Ergodicity	Measure tends to a limit when the number of fields of analysis increase
Repeatability		Negligible scatter of results obtained by several people using the same technique at given laboratory
Reliability - reproducibility		Negligible scatter of results obtained at different labs
Clarity and usefulness		Form of results presentation assuring: • ability to understand and interpret results without deep knowledge of quantitative metallography, • emphasizing diversification of the microstructure on transverse and longitudinal sections, • application of statistical analysis and methods of experimental research methodology for evaluation of structure - property relationships
Efficiency - economy		Measure of method's efficiency: efficiency index = 1 : (measurements time consumption $\times$ SRE)$^{1/2}$ higher for better methods

Table 6. Factors affecting intrinsic and extrinsic variability of quantitative microstructure analysis results.



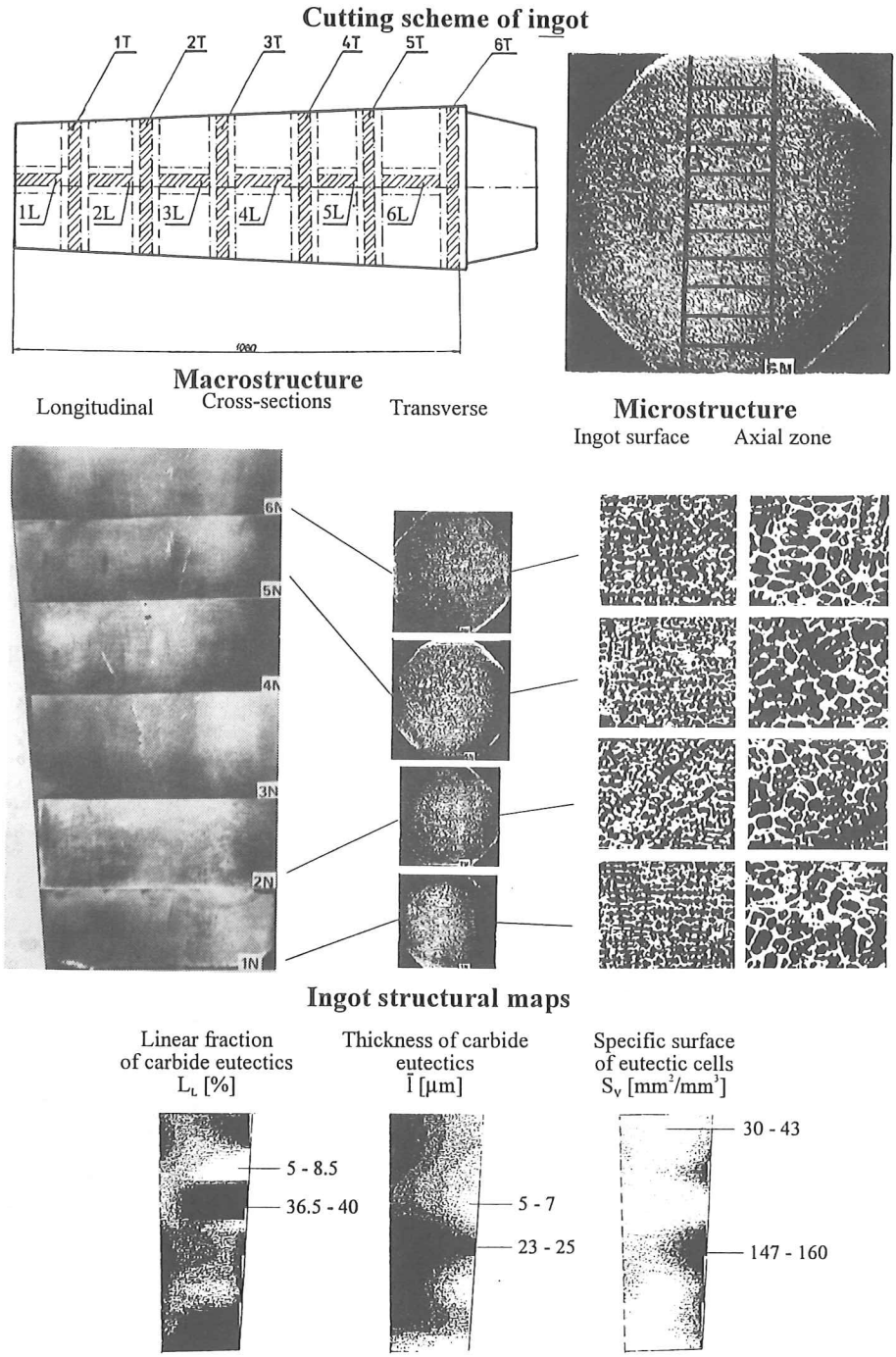


Fig. 2. Macro- and microstructure of SC 12-0-5 high-speed steel ingot (375/285x1080 mm - 900 kg).

A number of analysed fields in each microregion is a function of structure heterogeneity and number of measurements necessary to obtain assumed accuracy, according to the proposal (De Hoff, 1968):

$$N = [200 \cdot s(x)]:[RE[\%] \cdot \bar{x}]$$

where: s(x) - empirical standard deviation of the estimated parameter,
$\bar{x}$ - arithmetic mean of this parameter,
RE - assumed relative error.

In ASTM E 1245-89 standard it is recommended to apply 100 to 300 fields of analysis. In (Allmand and Coleman, 1971) the number of fields necessary to obtain with automatic structure analyser relative error not exceeding 5% was estimated as 2500 to 8400 in case of small inclusion content.

Labour demand and cost of experiments increase with the increase in assumed accuracy of measurements. Thus, applying both too small and too large number of measurements is the methodological mistake. When fixing necessary number of measurements one should take in mind an inherent microstructure feature of many rolled steel products - axially symmetrical inhomogeneity. The accuracy of the stereological parameters calculation should ensure that the difference between results obtained for the microregions selected at the same distance from the symmetry axis is insignificant. However, the essential microstructure diversification on the transverse cross-section should be infallibly revealed.

It has been shown that variation in results of quantitative structure assessment reaches the largest value among specimens, smaller among microregions and become the smallest among subsequent observation fields (Underwood and Starke, 1978).

Taking into account the above remarks concerning "intrinsic variability" one can state that Weibel's (1978) suggestion: "thinking and planning is more effective than labouring - do more less well" is particularly applicable in case of quantitative metallography.

The importance of preparation and observation techniques, methods of image analysis as well as measurement conditions for the accuracy of final results is illustrated in Table 6 and Figs 3÷4.

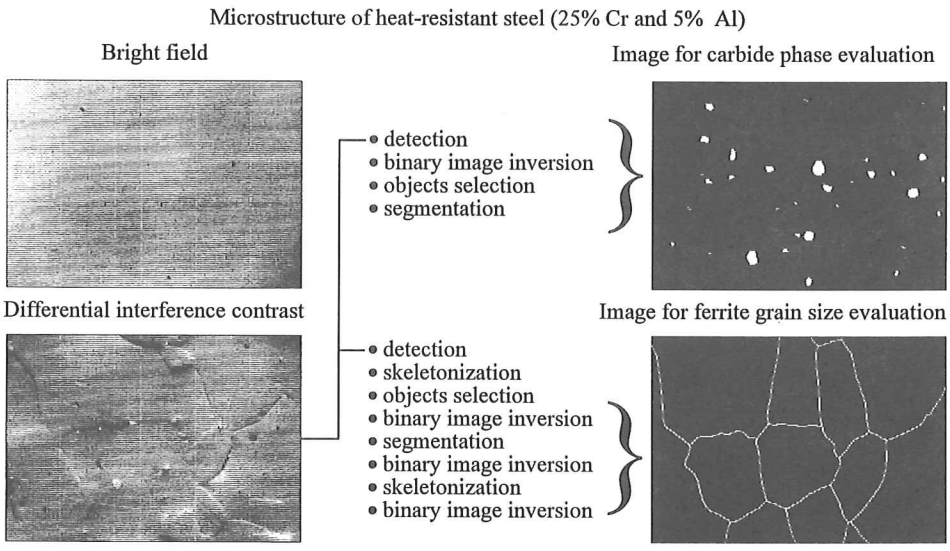


Fig. 3. Role of microscopic observation and image analysis methods in quantitative metallography of steel.

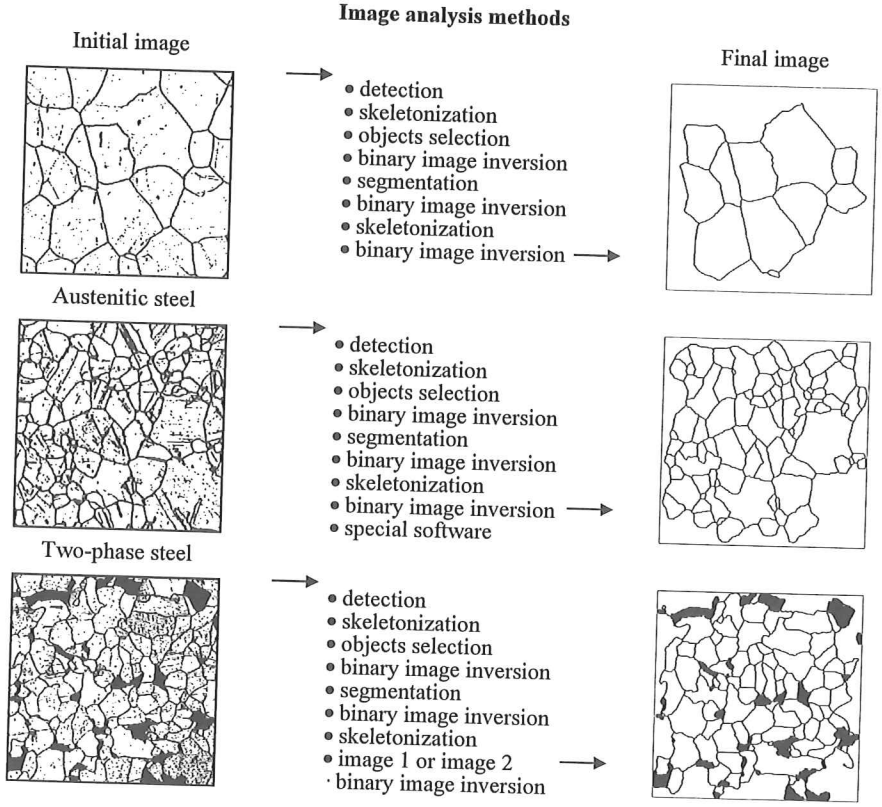


Fig. 4. Examples of image analysis methods application for an elimination of preparation artefacts and particles as well as for grain boundary reconstruction in steel granular microstructure examination.

The chosen method of analysis should ensure reproducible results what should be checked each time by appropriate statistical tests. In this range a significant improvement has been noticed recently. It is associated with the following factors:

- rapid and noteworthy progress in apparatus and abrasive materials for cutting, grinding and polishing of specimens as well as introducing expert systems which enable obtaining acquisition of polished sections from any steel with satisfactory quality (Bjerregaard et al., 1992),
- elaboration of new image analysis techniques like Fourier transformation, directional oriented mathematical morphology operations for binary and gray scale images enabling elimination of polished sections' defects and analysis of lamellar structures (Jeulin and Kurdy, 1992) as well as application of granulometry and granulomorphy in light and scanning electron microscopy (Chermant and Coster, 1990),
- increase in operating abilities of image analysers by introducing new hardware and software solutions.

It seems necessary to develop a separate branch of metallographic preparation and inspection oriented on the needs of quantitative metallography. It results from the essential difference in requirements: classical (qualitative) metallography tends to visualize all the existing structural constituents while in quantitative analysis only selected features, being currently analysed, should be emphasized. Methods

of selective etching, light microscopy in polarized light, with interference and phase contrast as well as application of special techniques in scanning electron microscopy can facilitate preparation of correct microstructure images suitable for measurements (Habraken and De Brouwer, 1968; Gifkins, 1980; Mc Call and French, 1979; Kunze, 1971).
The human factor, emphasized in Table 6, causes that structural criteria can posses their required properties (shown in Table 5) only if the whole testing procedure is thoroughly planned and consequently checked at every stage of specimen preparation and analysis process.
The above study points that stereological parameters of structure constituents can play the role of quality control criteria only if evaluated using automatic image analysers.

CONCLUSIONS

In recent years a significant progress in application of stereological methods in MSE is observed. It refers mainly to microstructure description in theory of hardening by grain refinement (Table 7), precipitation strengthening (Liu, 1992), coagulation (Ryś, 1986), fracture (Pacyna, 1988), creep-resistance (Hernas and Maciejny, 1989) and physical-chemical properties of steel (Łaskawiec, 1988). Quantitative methods are also gradually introduced into standards (JIS G 0555-1956 (JAPAN); BN-77/4054-01 (POLAND); ASTM E 1245-89, ASTM E 1222-86, ASTM E 562-89, ASTM E 1268-88, ASTM E 112-88, ASTM E 1181-87, ASTM E 930-83 (USA); DIN 50601 (FRG)).

Table 7. Examples showing the theory of grain boundary strengthening development.

Equation	Measure of grain size applied
1. Hall-Petch's equation - Petch (1953): $\sigma = \sigma_o + kd^{-0.5}$ σ - yield stress, σ_o, k - parameters dependent on temperature and deformation	d - mean grain diameter (various versions of Hall-Petch equation published in 1953 ÷ 1978)
2. Taylor-Sachs' theory developed by Dollar and Gorczyca (1984): $\sigma = \sigma_o + A_1\varepsilon^{n/2} + A_2\varepsilon^{(1-n/2)}S_V$ A_1, A_2, n - constants,	S_v - specyfic surface of grain boundaries
3. Kühlmeyer's (1979) equation: $\sigma = k\left[\frac{F_o \ln(2)}{\pi N \varphi(D_j) D_j^{3.5}} + \frac{1}{2\sqrt{D_j}}\right]$ F_o - area of specimen transverse cross-section, N - number of grains on "shear stress" plane	D_j - grain diameter, $\varphi(D_j)$ - grain diameter distribution
4. Bucki and Kurzydłowski (1991) empirical equation: $\sigma_p = \sigma_o + k_V\left[-\frac{9}{8}SD(V)^2\right]E\left(\sqrt[3]{V}\right)^{-1/2}$ σ_o, k_v - constants	E(V) - mean grain volume SD(V) - empirical standard deviation of grain volume
5. Liu's (1992) equation - HSLA steel strengthening: $\sigma_y = \sigma_1 + \sigma_s + \left[\sigma_d^2 + \sigma_p^2 + \left(k_{sub} l_{sub}^{-1/2}\right)^2\right]^{1/2} + k_g l^{-1/2}$ σ_y- yield stress, σ_s, σ_d, σ_p - solid solution, dislocation, precipitation strengthening, "g", "sub" - grain and subgrain boundary strengthening,	$\bar{l}$ - mean intercept length

These positive changes are, however, too slow. The main reasons of this state seem to be:

- low level of stereological knowledge among specialists involved in quality control and preparation of standards,
- lack of belief that switching to quantitative methods will improve the results of quality control to the level which will balance high cost connected with the new apparatus for specimen preparation and image analysis. Simultaneously, a significant number of well equipped image analysis laboratories at universities and industrial research centers shows that the problem lies in organization rather than in technical barriers,
- necessity to break up old habits and anxiety that all the experience, collected by years, will be lost. It is groundless as modern image analysers are equipped with software enabling correlation between comparative and quantitative methods.

To summarize it seems to be necessary to:

- prepare a handbook "Practice of stereological methods". It should be written after A.Einstein "As simply as possible but not more simply" since the available monographs are written mainly by stereologists for stereologists,
- elaborate for typical steel microstructures an atlas of morphological transformations necessary to perform automatic measurements,
- publish in main journals devoted to MSE a series of papers on current state of stereology,
- organize courses, workshops and conferences on application of stereological methods in MSE for materials engineers and quality control specialists,
- run under the auspices of ISS an international research program on "Sampling strategy and methodology of quantitative structure assessment in steels". The aim of this program would be demonstrating that current level of stereology and image analysis enables determination of objective structural criteria of steel quality at acceptable cost and labour demand,
- enhance the role of ISS members in national and international standard committees.

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