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Manufacturing technologies of sintered graded tool materials evaluated according to foresight methodology

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Abstract

Purpose: The goal of this chapter is to evaluate the development efficiency of conventional technologies of powder metallurgy used for graded tool materials manufacturing. The technologies were divided into three groups according to the matrix type and the percent fraction volume of components in the powders layers.

Design/methodology/approach: In the framework of foresight-materials science research a foresight matrices set was created, materials science experiments using light, transmission and scanning electron microscopes, X-ray diffractometer, microhardness tester, work-stands for testing of fatigue resistance, mechanical fatigue strength, fracture toughness were conducted and technology roadmaps were prepared.

Findings: Quite high potential and attractiveness of the analysed technologies against the environment, as well as good development perspectives in industry were shown.

Research limitations/implications: Research concerning graded tool materials constitute a part of a larger research project aimed at identifying, researching, and characterising the priority innovative technologies in the field of materials surface engineering.

Practical implications: The presented materials science results prove a manufacturing possibility of elements with ductile cores and hard coatings using conventional technologies of powder metallurgy. These technologies are recommended for practical implementation in industry, especially for cutting tools.

Originality/value: The originality of this chapter the value evaluation of manufacturing technologies of graded tool materials against background environment including the influence of the chemical composition and sintering conditions on the surface layers hardness.

Keywords: *Manufacturing and processing; Powder metallurgy; Graded tool materials; Foresight; Technology Roadmapping*

This chapter has been also published as:

A.D. Dobrzańska-Danikiewicz, A. Kloc-Ptaszna, B. Dołżańska, Manufacturing technologies of sintered graded tool materials evaluated according to foresight methodology, Archives of Materials Science and Engineering 50/2 (2011) 69-96.

1. Introduction

In line with the latest European development trends in the current, hard economic situation and the accompanying issues concerning the climate change, increased consumption and depletion of conventional energy sources, food safety, healthcare and the advancing ageing of the society, innovation understood as valuable, innovative ideas should be the way to achieve the set strategic objectives and represent the source of economic growth. Having this concept in mind, the European Commission has prepared a draft on the establishment of the Innovation Union [1], by focussing on the innovations that should substantially contribute to facing the key challenges named in the valid Europe 2020 strategy. The strategy provides that the level of R&D and innovation investments until 2020 is to reach aggregately 3% of the EU's GDP from public and private funds. For the economic and social effects achieved to be satisfactory, the stream of investments has to be channelled into those fields of science and industries bringing the highest added value, with special consideration given to the role of small- and medium-sized enterprises representing 99.8% of all the Polish enterprises generating 68% of the GDP. The aim of the foresight studies conducted widely in Europe and Poland in the recent decade, also for materials science [2-5], is a quest for innovative areas deserving financial support. Technology foresight serving to identify the priority, innovative technologies and their strategic development directions, has been undertaken for materials surface engineering, as well [6, 7]. Surface engineering of tool materials represents one of the 14 thematic areas analysed for these foresight studies. Of 10 critical technologies having the best development prospects and/or being of key significance for the industry selected for thorough research

carried out with the Delphi method, the powder metallurgy methods were found allowing to achieve changes to chemical composition and/or phase composition in the surface layer.

Graded tool materials, characterised by their chemical composition, phase composition and the structure or arrangement of atoms varying according to the location, can be produced in laser treatment (remelting and alloying) [8-11], Physical Vapour Deposition (PVD) [12-18], hybrid (PVD and thermochemical treatment) [19-22] and sintering processes with the conventional powder metallurgy methods [23-27]. This chapter discusses the last of the mentioned groups of manufacturing the graded tool materials. The production of graded tool materials in the conventional powder metallurgy method consists of filling a die with the subsequent layers of powder mixtures differing in their chemical and/or phase composition according to the required properties of the material produced with the mixtures being then pressed and sintered [28-31]. The output composition of the moulded piece can, therefore, be controlled in order to produce sinter with its chemical and/or phase composition varying according to the material volume and position. Such materials are called Functionally Graded Materials (FGM) [32-35]. If the powder metallurgy method is applied, high resistance to abrasive wear, distinctive for cemented carbides and cermets, can be accomplished for the surface layer while maintaining high core ductility typical for high-speed steels [24-27, 36] and traditional carbide steels [37-40] at a relatively low cost. Such structure of the material allows to develop its properties freely depending on the working conditions of the tool. A layer of hard material is used for example in those locations at the tool being exposed to wear while high ductility is ensured in those locations of the tool material that are susceptible to impact.

The purpose of this study is a comparative analysis of three specific technologies of producing the following graded tool materials with the conventional powder metallurgy method: GM-90HSS/10WC, GM-75HSS/25WC, GM-3Co/97WC. Categorisation is done according to the criterion of the matrix material and the fraction of powder in the mixture. The subject of the comparative analysis are both, the outcomes of investigations into the structure and properties of the analysed materials as well as the value of the individual technologies against the environment and their long-term development prospects together with the recommended management strategies and forecast multi-variant development tracks with this value being determined through expert studies according to the custom methodology [41]. The relevance and adequacy of the assessments performed is ensured by the synergic interaction and cross supplementation of the foresight-materials science research. The chapter

also presents the outcomes of e-foresight research (Fig. 1), based on row data [6] pertaining to the position of tool materials surface engineering, including the powder metallurgy methods, permitting to change chemical and/or phase composition in the surface layer, vis-à-vis other surface engineering technologies. Technology roadmaps being a comparative analysis tool especially helpful for the small- and medium-sized enterprises lacking funds for conducting own research in this domain have been established at the last stage of the efforts. The results of the foresight and materials science research presented in this chapter are part of a broader project [7] aimed at selecting the priority innovative technologies of materials surface engineering and setting their directions of strategic development, as discussed in a series of publications, *inter alia* [42-47].

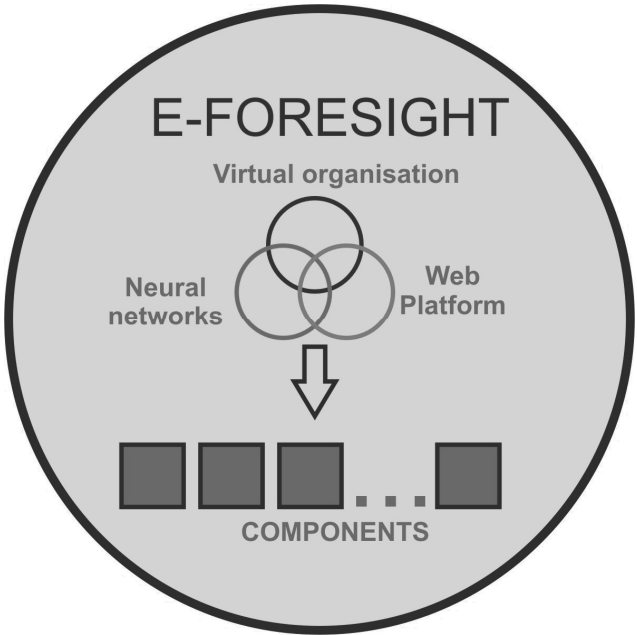


Figure 1. E-foresight research

2. Subject matter of interdisciplinary research

The research pursuits concentrated on graded tool materials, differing in the matrix material and a volume fraction of components in the individual layers of powders, are of an interdisci-

plinary character. The research methodology applied concerns primarily technology foresight (Fig. 1) being part of the field of science referred to as organisation and management and of surface engineering comprised in the widely-understood materials science. A much broader insight into the concept was, however, required at some stages of the research, hence the following areas of detailed knowledge were also derived from: information science with information technology and artificial intelligence [48, 49] (neural networks, Monte Carlo method); statistics; econometrics; operational studies; construction and operation of machines; automation and robotisation of industrial processes; strategic, tactical and operational management; quality and environment management; accounting and finance.

The conventional powder metallurgy method has been used for producing the materials. The method consists of the uniaxial pressing of powder in a closed matrix and then sintering it. The graded materials were produced by filling the matrix with the subsequent layers of powders mixtures and next by pressing and sintering the layers. The following three homogenous specific technologies were distinguished between for the purpose of the foresight and materials science works carried out under this study by adopting, as a criterion of grouping, the matrix material and the volume fraction of components in the individual layers of powders:

- (A) Manufacturing of the GM-90HSS/10WC graded tool materials based on the high-speed steel matrix with 10% volume fraction of the tungsten carbide reinforcing phase in the surface layer,
- (B) Manufacturing of the GM-75HSS/25WC graded tool materials based on the high-speed steel matrix with 25% volume fraction of the tungsten carbide reinforcing phase in the surface layer,
- (C) Manufacturing of the GM-3Co/97WC sintered graded carbide steels based on the cobalt matrix with 97% volume fraction of the tungsten carbide reinforcing phase in the surface layer.

2.1. Test materials

The research has been carried out with specimens manufactured with a conventional powder metallurgy method. The powders of HS6-5-2 high-speed steels (Fig. 2), tungsten carbide (Fig. 3) and cobalt (Fig. 4) have been used for the research. The chemical composition and the basic properties of powders are juxtaposed in Table 1.

Table 1. Chemical composition and properties of HS6-5-3, WC and Co powders

Element	Mass concentration, %		
	HS6-5-2	WC	Co
C	0.75-0.90	6.11	0.02
Mn	0.20-0.45	-	< 0.001
Si	≤ 0.45	≤ 0.002	< 0.002
P	≤ 0.04	-	-
S	≤ 0.04	0.003	< 0.002
Cr	3.75-4.5	-	-
Ni	0.2	-	< 0.002
Mo	4.5-5.5	≤ 0.001	-
W	5.50-6.75	rest	-
V	1.6-2.2	0.19	-
Co	0.1	-	rest
Cu	0.1	-	< 0.002
Fe	rest	0.003	-
Ca	-	0.003	< 0.001
Al	-	≤ 0.002	-
Mg	-	≤ 0.001	-
K	-	≤ 0.001	-
Na	-	≤ 0.001	-
C free	-	0.02	0.02
Grain size, μm	> 150	> 0.86	> 6
Additional information	High-speed steel powder, atomised with water by Hoeganaes	Tungsten carbide powder by Unicore	Cobalt powder by Unicore

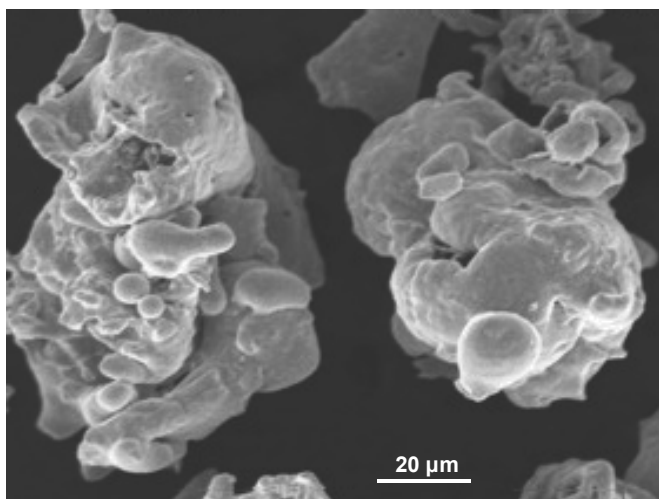


Figure 2. A scanning electron micrograph of HS6-5-2 water atomised powders

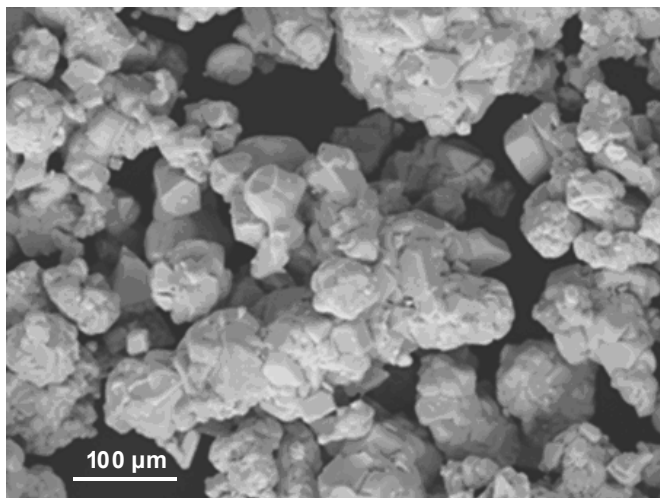


Figure 3. A scanning electron micrograph of WC powders

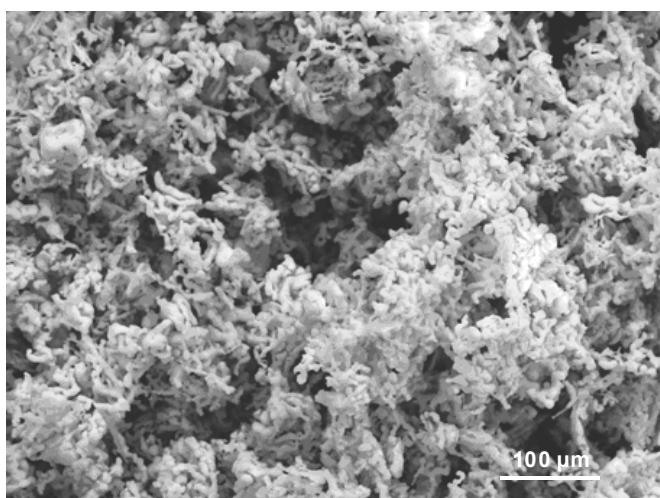


Figure 4. A scanning electron micrograph of Co powders

According to the (A) and (B) technologies four-layer specimens were made of HS6-5-2 high-speed steel powder and WC powder. These specimens were made where the subsequent transitory layers with a smaller and smaller volume fraction of tungsten carbide were established from the surface layer side until a surface layer was produced containing high-speed steel only. An assumption was made that such combination of layers can correspond

to the material used for producing tools for cutting edges. The powders were mixed for an hour in a T2F WAB-TURBULA mixer. The powder mixtures were next filled to the matrix thus producing layers with the gradually changing volume fraction of carbides in high-speed steel. The next transitory layers were established according to the (A) technology for the volume fraction in the surface layer of 10% of WC containing, respectively, 7 and 4% of such carbides. In the case of the (B) technology, where the surface layer containing 25% of the WC fraction volume, the transitory layers contained respectively, 15 and 5% of WC. The specimens were pressed under the pressure of 500 MPa. The sintering conditions were selected experimentally by changing a temperature, duration and atmosphere of mouldings sintering. The specimens were sintered in a vacuum furnace and with the atmosphere of flowing nitrogen with the addition of hydrogen ($N_2+5\% H_2$), at a temperature of 1210, 1230, 1250 and 1270°C, for 30 and 60 minutes.

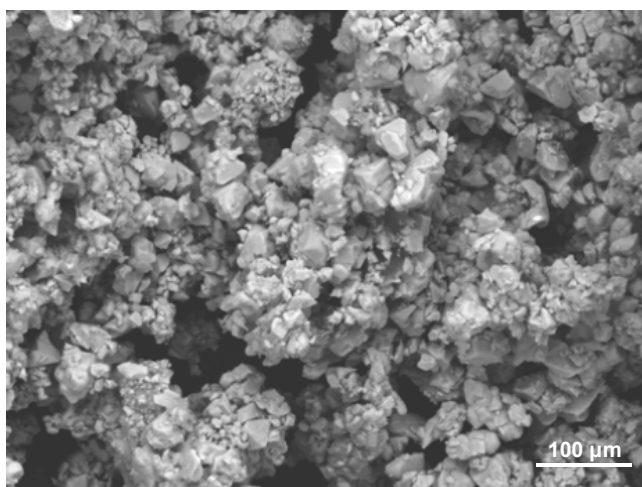


Figure 5. Mixture of WC (93%) and Co (7%) powder after 8 hours of crushing in a ball mill

According to the (C) technology four-layer specimens were made of tungsten carbide powders (WC) and cobalt powder. These specimens were made with the individual transitory layers being established in the surface layer side with an increasingly higher volume fraction of cobalt carbide until a layer has been produced containing 9% of Co and 91% of WC. An assumption was made that such combination of layers can correspond to the material designed for producing tools for cutting edges. Co and WC powders were crushed in a ball mill

with carbide balls for 8 hours (Fig. 5). The powder mixtures obtained were next filled to the matrix thus producing layers with the gradually changing volumetric concentration of cobalt and tungsten carbide fraction. The subsequent four transitory layers were established in the surface layer of the material with the volume concentration of 3% of Co and 97% of WC with a 2% increase of cobalt concentration until a surface layer was produced containing 9% of Co and 91% of WC. The mouldings were produced as a result of pressing under the pressure of 340 MPa. The sintering conditions were selected experimentally by changing a temperature, duration and atmosphere of mouldings sintering [50,51]. The specimens were sintered in a vacuum furnace, freely at a temperature of 1460°C, for 30 minutes and with isostatic pressing at 1425°C, for 90 min.

Table 2 shows the detailed marking of the specimens and their layers with the volume fraction of mixture components provided for the individual layers.

Table 2. The detailed marking of the GM-HSS/WC and GM-Co/WC specimens and their layers with fraction volume of components in the layers

Specimen marking*)	Layer marking*)	Layer number	Layer type	Fraction volume of components in layers, %					
					Powder		Powder		
GM-90HSS/10WC	GM-90HSS/10WC	1	Surface layer	90	HS6-5-2	10	WC		
	GM-93HSS/7WC	2	Intermediate layers	93		7			
	GM-96HSS/4WC	3		96		4			
	GM-100HSS	4	Substrate	100		0			
GM-75HSS/25WC	GM-75HSS/25WC	1	Surface layer	75		Co		25	WC
	GM-85HSS/15WC	2	Intermediate layers	85				15	
	GM-95HSS/5WC	3		95				5	
	GM-100HSS	4	Substrate	100				0	
GM-3Co/97WC	GM-3Co/97WC	1	Surface layer	3	Co	97	WC		
	GM-5Co/95WC	2	Intermediate layers	5		95			
	GM-7Co/93WC	3		7		93			
	GM-9Co/91WC	4	Substrate	9		91			

*) GM – graded material; HSS – high-speed steels; WC – tungsten carbide; Co – cobalt

2.2. Materials science methodology

In the framework of this chapter have been carried out research of structure as well as mechanical and physical properties of GM-HSS/WC and GM-Co/WC manufactured by the conventional powder metallurgy method.

Metallographic research has been performed on the fractures of the sintered and heat treated specimens. The specimens were cut in a plane perpendicular to the surface layer. Metallographic research has been conducted with light microscopes (MEF4A by Leica and Axiovert by OPTON) with the magnification of 100 to 1000x and with scanning electron microscopes (XL30 by Philips Company and Supra 35 by Zeiss) fitted with secondary electrons detectors (SE) and back-scattered electrons detectors (BSE or QBSD) with the accelerating voltage of 5 to 20 kV, with the magnification of 50 to 10000 times. The primary austenite grain size was measured with the Snyder-Graff method. The fraction volume of carbides was determined with the methods of quantitative metallography using Image-Pro Plus computer aided image analysis software.

The quantitative and qualitative X-ray microanalysis and the surface distribution analysis of alloy elements was performed on the ground and polished fractures from the surface layers of gradient sintered carbide steels in a scanning electron microscope (XL30 by Philips Company), at the accelerating voltage of 20 kV, equipped additionally with an X-ray radiation analyser with energy dispersion (EDAX D4) by Philips Company. The chemical composition (mass concentration and atomic concentration of metallic elements) was determined with an X-ray microanalysis in the selected micro-areas of the matrix and carbides on the cross-section of the investigated graded tool materials.

The phase composition of powders, mouldings and graded materials in the sintered and heat-treated condition was evaluated with an X-ray X'PertPRO diffractometer by PANalytical using filtered $K\alpha$ radiation of a cobalt lamp with the rated voltage of 40 kV and the filament current of 30 mA. Deflected radiation concentration measurements were carried out within the 2θ angular range of 30 to 120° with a 0.05° step and with the counting time of 10 seconds.

The fraction volume of residual austenite was calculated using the software developed at the Division of Materials Processing Technologies and Computer Techniques in Materials Science. The software enables fraction volume calculations for residual austenite in steels with an X-ray Averbach-Cohen method based on the results of measurements of total concentrations of diffraction maximums for X-ray radiation from the lattice planes of γ and α phase.

Diffraction investigations and the investigations of thin foil structures from the selected locations of the graded material specimens in the sintered and heat-treated condition were undertaken with a Transmission Electron Microscope (TEM) JEM 3010UHR transmission electron microscope by JEOL, with the accelerating voltage of 300 kV. Diffraction patterns from the TEM were solved using Eldyf software.

The proper (actual) density of high-speed steel and WC powders was measured automatically with a gas AccuPyc 1330 pycnometer by Micromeritics. The measurements were made in a helium atmosphere. The density, porosity and hardness was measured for the sintered specimens with the Vickers method and the Rockwell method at the A scale. A densitometric method was used for measuring the density of sinters. The method consists in measuring the apparent loss of the specimens mass when submerged in water. A hardness measurement was performed with the Vickers method with the indenter load of 4.903 N. The working time of the indenter total loading force was 15 seconds. The measurement was made across the entire width of the sintered specimens cross-section by starting the measurement within 0.2 mm from the outer surface layer and ending in the surface layer area (about four measuring points were used for each layer). Hardness testing with the Rockwell method at the A scale was made at the faces of the surface layers of the sintered gradient carbide steels. The measurement was made in 10 randomly points selected from the surface layers areas. Stereological tests using an Axiowert 405M optical microscope fitted with computer aided image analysis software were made to identify porosity. The tests were made at the fractures using Image-ProPlus computer-assisted image analysis software. The surface fraction of pores was determined – expressed as a ratio of the pores area to the total area of the analysed area. Five random points from the areas of the individual layers were chosen each time for measurement. Hardness tests with the Rockwell method at the C scale were made at the faces of the surface layers of graded materials on a heat-treated high-speed steel matrix. A measurement was made for each heat treatment variant in the randomly picked points from the area of surface layers and substrate (10 measurements for each layer). The results of density, porosity and hardness tests were developed statistically by calculating, for each series of measurements, the average value, standard deviation and average value confidence interval for the confidence level of $\alpha = 0.05$. A regression function was also determined approximating the relationship between the investigated output variable (e.g. material hardness or density) and the input variables (e.g. volume fraction of components, production or thermal treatment process conditions).

The tests of abrasive wear resistance for GM-3Co/97WC were carried out using the device shown in Fig. 6. Tests with a counter-specimen, i.e. an Al_2O_3 ceramic ball were made on the prepared specimens. The tests were made using a varied number of 1000 cycles and at a varied load of 2.5 and 10 N. Four results were obtained for the surface layers of each tested specimen due to such applied tests conditions and this allowed to define an abrasive wear measurement. The degree of wear was determined through the geometric measurements of wear and by calculating the wear volume. Wear observations were also made with a Confocal Laser Scanning Microscope (CLSM) 5 Exciter confocal microscope and in an Scanning Electron Microscope (SEM).

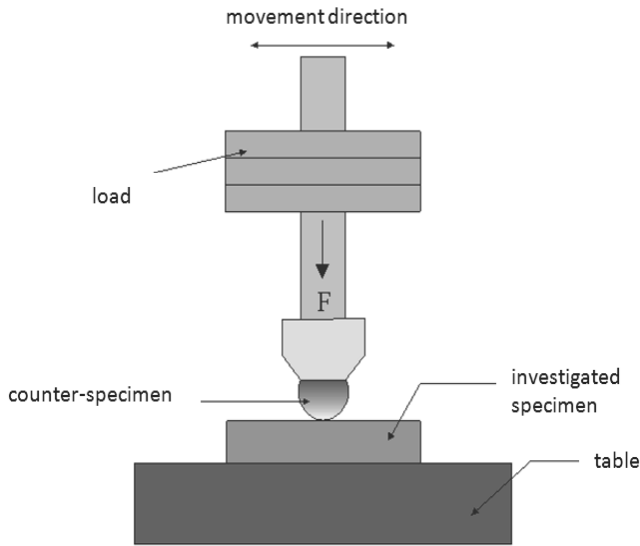


Figure 6. Diagram of the abrasive wear resistance testing device

Resistance to brittle cracks (K_{IC}) was tested according to [52] using the Palmqvist method (Fig. 7). The tests were made at the appropriate samples prepared in advance.

The following formulae were used to determine the K_{IC} co-efficient:

$$H = \frac{1.854 \times P}{\left[(d_1 + d_2) \times \frac{1}{2} \right]^2}, \quad \text{N/mm}^2, \quad (1)$$

where:

P – load applied, N,

d_1, d_2 – length of the indentation diagonal, mm,

$$T = l_1 + l_2 + l_3 + l_4, \quad (2)$$

where:

T – sum of cracks length, mm,

$$K_{IC} = A\sqrt{H} \times \sqrt{\frac{P}{T}}, \text{ MNm}^{-3/2}, \quad (3)$$

where:

A – constant 0.0028.

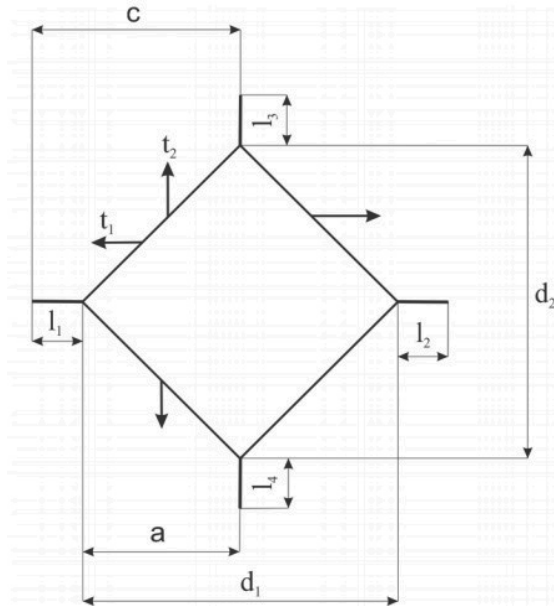


Figure 7. Diagram of cracks system obtained with the Palmqvist method

During investigations accept traditional material science methods also the Finite Element Method (FEM) was used. Next, the research results were experimentally verified using the X-ray spectrometer. FEM was used for performing a computer simulation of gradient stresses of carbide tool materials [25, 53], internal stresses and material work deformations. The actual model of the graded tool material was designed with Inventor 11 software and the strength analysis was performed with ANSYS 12.0 software. The following boundary conditions were assumed to simulate the internal stresses of the graded tool material:

- a variation in the sintering temperature is reflected by the specimens cooling process from 1460°C to the ambient temperature of 22°C,

- material properties for the material produced were assumed based on the product sheets of the MatWeb catalogue provided in Table 3.

Table 3. Mechanical and physical properties assumed for the computer simulation of internal stresses occurring in the produced material made up of four layers with a varied fraction of tungsten carbide and a varied cobalt concentration [54]

Properties	Four layers of GM-Co/WC			
	3% Co+97% WC	5% Co+95% WC	7% Co+93% WC	9% Co+91% WC
Young modulus, GPa	665	640	615	590
Poisson co-efficient	0.2809	0.2815	0.4774	0.5338
Density, g/cm ³	15.4	15.1	14.8	14.5
Thermal expansion, 10 ⁻⁶ 1/K	4.1	4.3	4.5	4.7
Heat conductivity, W/(m·K)	98	90	82	76
Specific heat, J/(kg·K)	138.7	144.5	150.3	156.1
Resistivity, Ω·m	5.4252	5.442	5.4588	5.4756
Tensile strength, MPa	1670.75	1641.25	1611.75	1580.25

A model for determining the internal stresses of tool work was prepared using the finite element method by assuming the actual specimens dimensions. The actual model was subjected to discretisation. The calculation model comprises 4968 nodes and 760 elements. The actual internal stresses in the tested materials were calculated in order to verify experimentally the results achieved by means of the modelling of the finite element method based on the measurements made using X-ray spectrometry. The calculations were made with the $\sin^2\psi$ method with the custom X'Pert Stress Plus software. The software incorporates data, as a database, necessary to calculate the values of material constants. A comparative analysis of computer simulations to the experimental results was then made.

2.3. Foresight methodology

The own reference data gathered when implementing the project “Foresight of surface properties formation leading technologies of engineering materials and biomaterials. FORSURF” [6] was used in order to determine the strategic position of the thematic area “Surface engineering of tool materials” against materials surface engineering and the manufacturing technologies of graded tool materials with the powder metallurgy methods in relation to other

tool materials manufacturing methods. Nearly 300 independent foreign and domestic experts representing scientific, business and public administration circles have taken part in the foresight research up till now at the different stages of the works. The experts have completed approx. 600 multi-question surveys and held thematic discussions during 7 Workshops. The initial phase of the foresight research, including the evaluation of the steady state, technology review and strategic analysis with integrated methods, was conducted for about 500 groups of detailed materials surface engineering technologies. The following scientific and research methods were applied at this stage: extrapolation of trends, environment scanning, STEEP analysis, SWOT analysis, expert panels, brainstorming, benchmarking, multi-criteria analysis, computer simulations and modelling, econometric and statistical analysis. 10 critical technologies with the best development prospects and/or of crucial importance for the industry in the next 20 years were chosen from each of the 14 thematic areas analysed as a result of the research performed. A collection of 140 critical technologies were thoroughly analysed according to three iterations of the Delphi method carried out in consistency with the idea of e-foresight [55, 56] using information technology encompassing a virtual organisation, web platform and neural networks. Neural networks were used in a novel and experimental manner to analyse the cross impacts between events to determine how growth, stabilisation or a decrease in the importance of the relevant factors of the analysis will contribute to the occurrence of each of the three anticipated scenarios at a macro scale. The relevant manufacturing technologies of sintered graded tool materials analysed in this chapter were evaluated based on the opinions of the key experts using the custom foresight-materials science research methodology [41]. The weighted scores method was used for a comparative evaluation aimed at classifying the importance of specific objects in the context of relationships between them. The approach adopted requires that the principle of relativising the evaluation criteria has to be applied, i.e. differences are assumed in the relevance of the criteria used and the principle of admissibility assuming that there is a certain set of admissibility conditions representing a selection filter classifying a certain object positively or negatively [57]. The weighted scores method enables to make a multi-criteria aggregate evaluation using a scale with intervals. A single-pole positive scale without zero, referred to as a universal scale of relative states, was used in the foresight research undertaken, where 1 is a minimum rate and 10 an extraordinarily high rate. The results of the expert evaluation of the technology for its potential, representing a realistic objective value of the particular technology, and for its attractiveness, reflecting the subjective perception of a specific technology by its potential users, were entered into one of the quarters

of the **dendrological matrix of the technology value**. Wide-stretching oak is the most promising quarter guaranteeing the future success. A quarter of soaring cypress groups highly attractive technologies with a limited potential and rooted dwarf mountain pine symbolises technologies with a high potential and limited attractiveness. If an appropriate strategy is used in both cases for a specific technology, its further progress and a robust strategic position in the future is feasible. The technologies with their success being either unlikely or impossible are placed in the field of quaking aspen. **The metrological matrix of environment influence** presents graphically the results of evaluating the influence of external positive factors, i.e. opportunities and difficulties, on the technologies analysed. Each of the technologies assessed by the experts was entered into one of the matrix quarters. Sunny spring illustrates the most favourable external situation ensuring the future success. Rainy autumn, providing a chance for steady progress, corresponds to a neutral environment and hot summer to a stormy environment where the success of a technology is risky but feasible. Frosty winter informs that adverse factors exist mainly with technology development being difficult or unachievable. The results of the expert investigations visualised with the dendrological and meteorological matrix were at the next stage of scientific work entered into the **matrix of strategies for technologies** made up of sixteen fields by means of software based on pre-defined mathematic relationships [41]. The matrix presents graphically a position of each technology according to its value and environment influence intensity and identifies a recommended action strategy. The strategic development prospects of a given technology expressed in numbers correspond to the relevant areas of the matrix. **Strategic development tracks** were also entered into the matrix of strategies for technologies reflecting the anticipating growth of the technologies analysed for the three variants: a positive, neutral or negative ones in the nearest 20 years for the time intervals of 2015, 2020, 2025 and 2030.

3. Materials science research results

3.1. Sintering conditions and the reinforcing phase fraction versus GM-HSS/WC properties

Figures 8 to 10 present the Regression Function Plots (RFP) describing a relationship between the tungsten carbide reinforcing phase density and fraction volume, sintering temperature and

sintering time for materials in a vacuum furnace and Figs. 11 to 13 for the furnace with the atmosphere of flowing nitrogen with addition of hydrogen ($N_2+5\% H_2$). It was found based on the results of density measurements for the sintered gradient carbide steels that the sintering temperature and atmosphere have a substantial effect on the density values. The sintering time, therefore, does not have a major effect on the density of the tested materials. The density of gradient carbide steels is between 6.4 to 8.3 g/cm^3 and between 7.5 to 8.6 g/cm^3 , respectively for GM-90HSS/10WC and GM-75HSS/25WC.

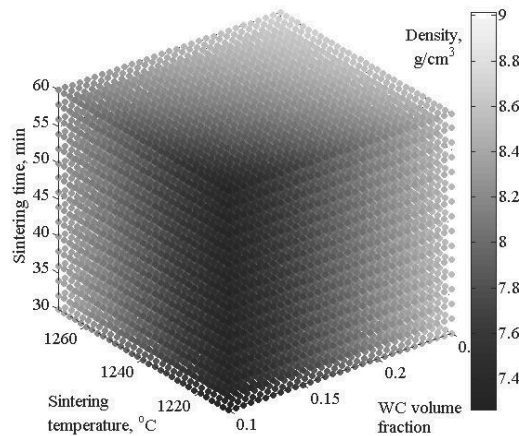


Figure 8. RFP describing relationship between density and the WC volume fraction, temperature, and sintering time, for GM-HSS/WC sintered in a vacuum furnace

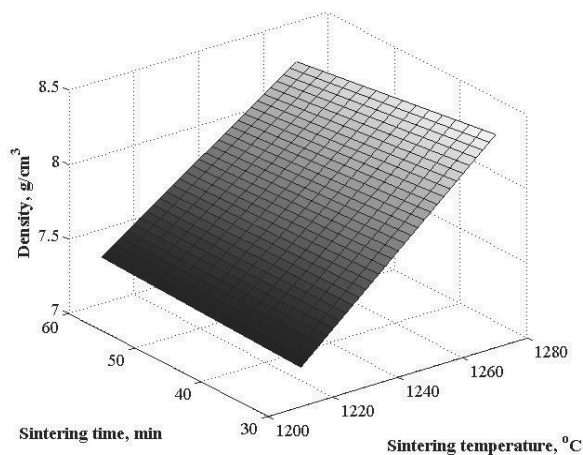


Figure 9. RFP describing relationship between density and the temperature, and sintering time, for GM-90HSS/10WC, sintered in the the vacuum furnace

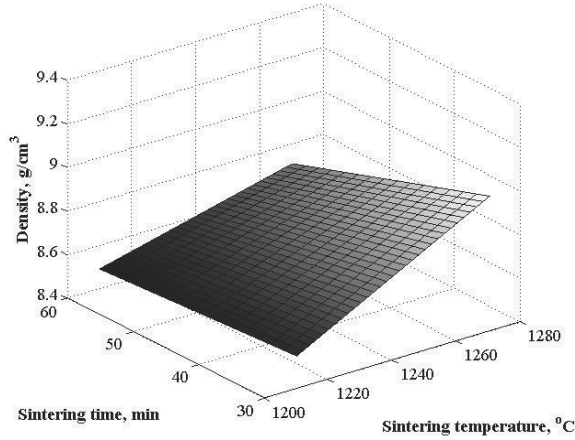


Figure 10. RFP describing relationship between density and the temperature, and sintering time, for GM-75HSS/25WC, sintered in the the vacuum furnace

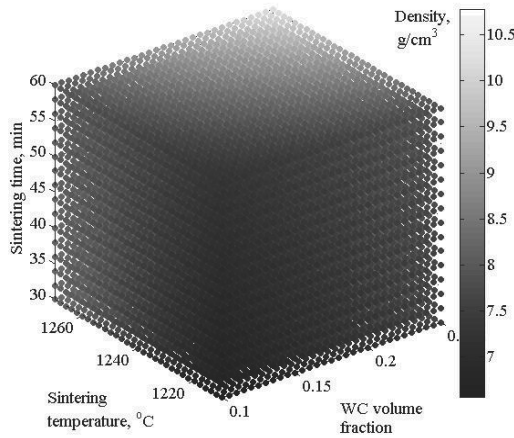


Figure 11. RFP describing relationship between density and the WC volume fraction, temperature, and sintering time, for GM-HSS/WC, sintered in furnace with the atmosphere of the $N_2+5\% H_2$

Figures 14 to 16 show the regression function plots describing the relationship between HRA hardness and the reinforcing phase volume fraction, sintering temperature and sintering time for materials in a vacuum furnace and Figs. 17 to 19 for the furnace with the atmosphere of flowing nitrogen with addition of hydrogen ($N_2+5\% H_2$). The results of hardness measurements for the surface layers of the tested materials sintered in a vacuum furnace and with the atmosphere of flowing nitrogen with an addition of hydrogen indicate that the sintering conditions and the fraction of the applied reinforcing phase significantly influence the

hardness of graded materials. The hardness of surface layers for the tested materials is within 55.7-80.8 HRA for GM-90HSS/10WC and 64.7-84.2 HRA for GM-75HSS/25WC. Moreover, no significant impact of the type of the applied sintering atmosphere on the tested materials hardness has been identified. As the temperature and volume fraction of WC is growing, so is growing the hardness of such materials surface layers. Variations in the sintering time within the sintering temperature of 1210 to 1270°C do not cause substantial changes to the hardness of the graded materials. The maximum surface layer hardness of approx. 84.2 HRA was achieved for GM-75HSS/25WC sintered in a vacuum furnace at a temperature of 1230°C for 30 min.

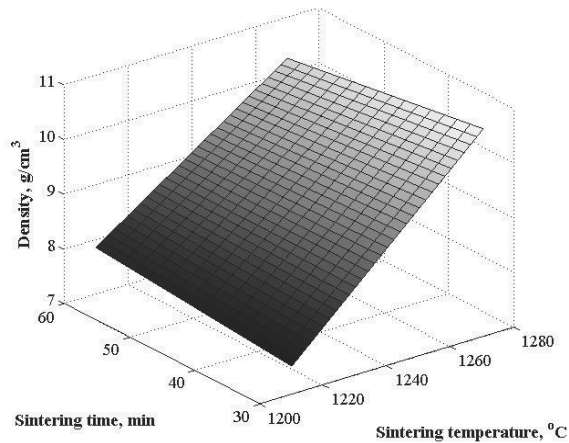


Figure 12. RFP describing relationship between density and the temperature, and sintering time, for GM-90HSS/10WC, sintered in furnace with the atmosphere of the $N_2+5\% H_2$

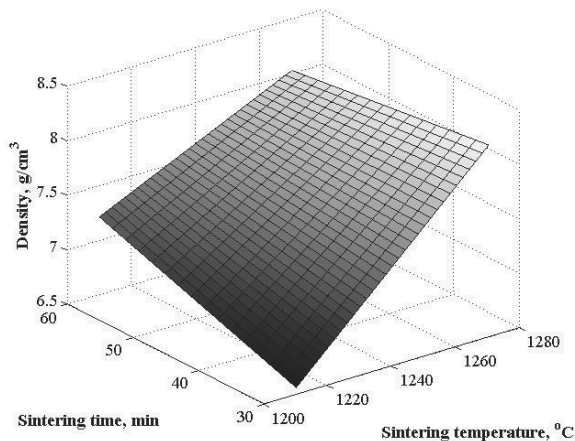


Figure 13. RFP describing relationship between density and the temperature, and sintering time, for GM-75HSS/25WC, sintered in furnace with the atmosphere of the $N_2+5\% H_2$

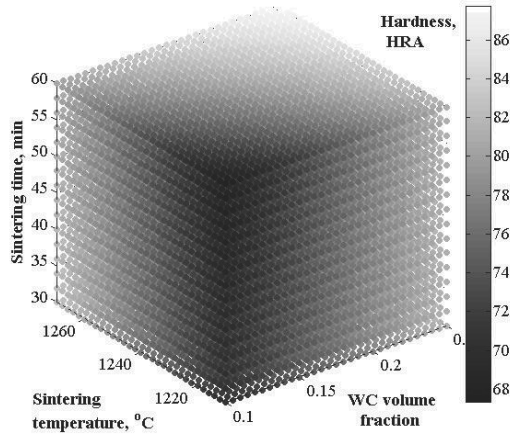


Figure 14. RFP describing relationship between hardness and WC volume fraction, temperature, and sintering time, for GM-HSS/WC sintered in a vacuum furnace

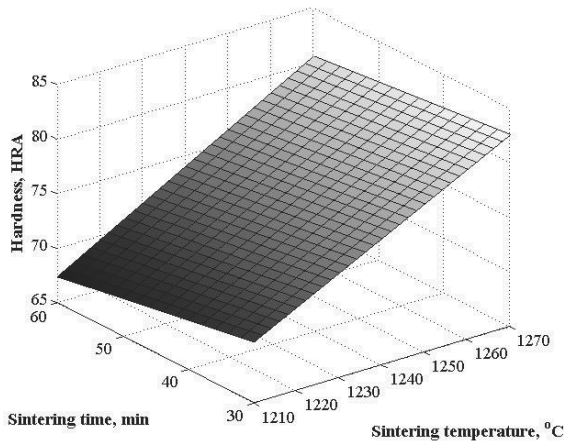


Figure 15. RFP describing relationship between hardness and the temperature, and sintering time, for GM-90HSS/10WC sintered in a vacuum furnace

Figures 20 to 21 illustrate the regression function plots describing the relationship between HV hardness and the measuring point location, sintering temperature and sintering time for materials in a vacuum furnace and Figs. 22 and 23 for the furnace with the atmosphere of flowing nitrogen with addition of hydrogen ($N_2+5\% H_2$). The results of HV hardness measurements show a gradient variation in the properties of the tested materials in their volume. The hardness value of all the tested materials, regardless the sintering conditions, changes along with the changing distance of the measuring point from the outer surface of the surface layer.

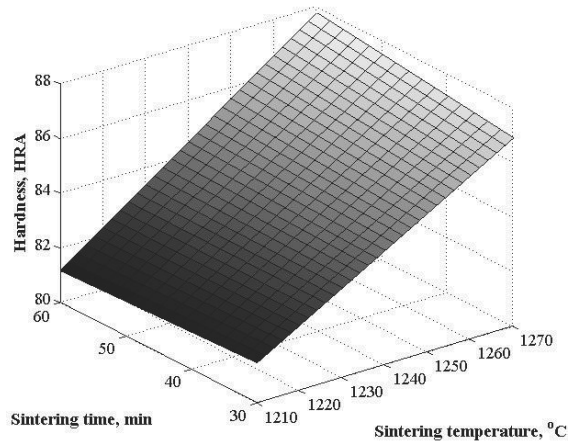


Figure 16. RFP describing relationship between hardness and the temperature, and sintering time, for GM-75HSS/25WC sintered in a vacuum furnace

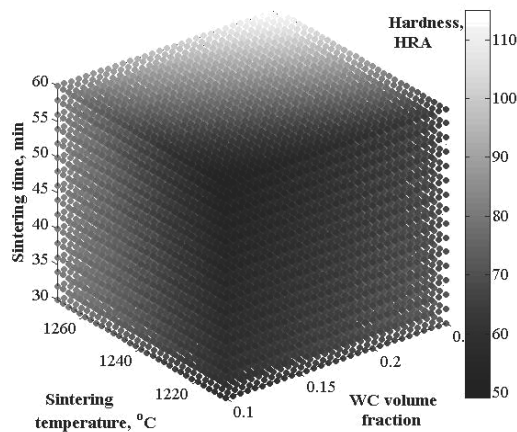


Figure 17. RFP describing relationship between hardness and WC volume fraction, temperature, and sintering time, for GM-HSS/WC, sintered in the furnace with the atmosphere of the flowing $N_2 + 5\% H_2$

The hardness of GM-90HSS/10WC sintered in vacuum depending on the sintering temperature is within 500-800 HV in the surface layer and decreases along with the growing distance between the measuring point and the outer surface of the surface layer up to 270-510 HV in the substrate layer. The hardness of GM-90HSS/10WC sintered in the atmosphere of the flowing gas mixture ($N_2 + 5\% H_2$) is within 580-680 HV in the surface layer and also declines along with the growing distance of the measuring point from the outer surface of the surface layer up to 350-540 HV. In GM-75HSS/25WC where the fraction of the WC reinforcing fraction in the

individual layers is higher (25% of WC in the surface layer), a hardness variation increases by approx. 100 HV in the surface layer. For GM-75HSS/25WC sintered in vacuum, it is within the range of 600-900 HV and decreases to 250-470 HV, and for GM-75HSS/25WC sintered with the atmosphere of the flowing mixture of gases, it is 300-860 HV in the surface layer and decreases along with the growing distance of the measuring point from the outer surface of the surface layer to 240-510 HV in the substrate layer.

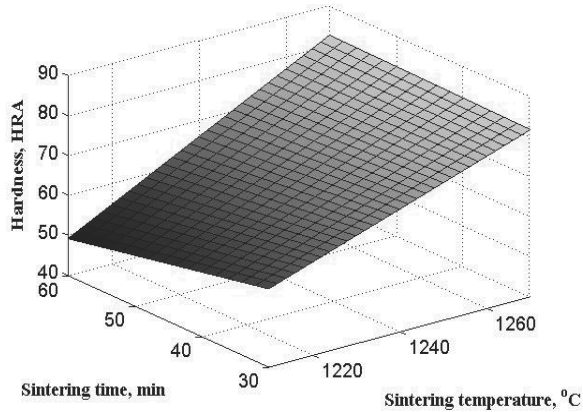


Figure 18. RFP describing relationship between hardness and the temperature, and sintering time, for GM-90HSS/10WC, sintered in the furnace with the atmosphere of the flowing $N_2+5\% H_2$

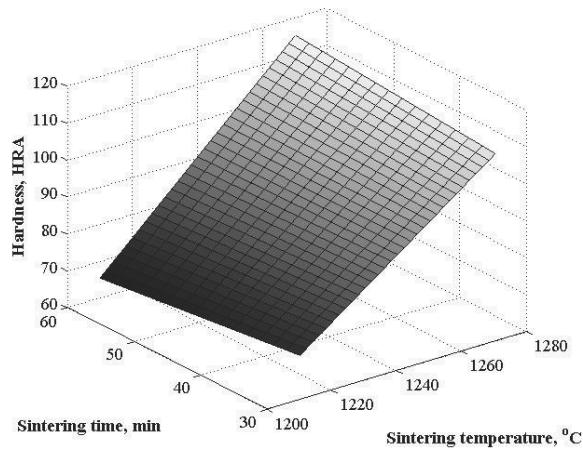


Figure 19. RFP describing relationship between hardness and the temperature, and sintering time, for GM-75HSS/25WC, sintered in the furnace with the atmosphere of the flowing $N_2+5\% H_2$

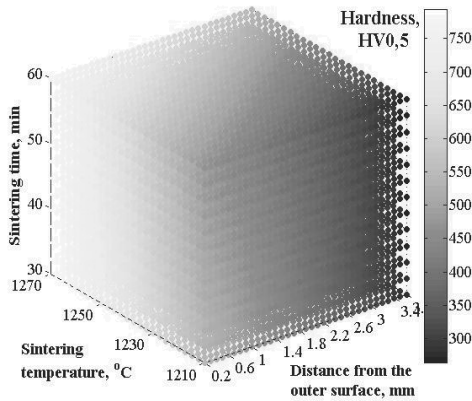


Figure 20. RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-90HSS/10WC sintered in a vacuum furnace

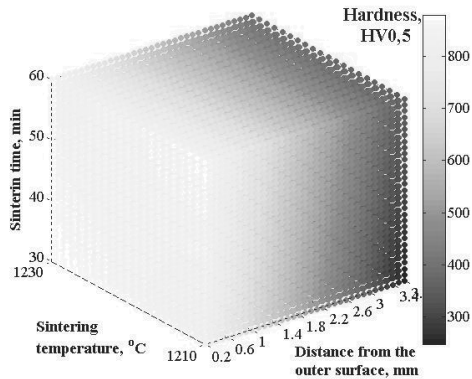


Figure 21. RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-75HSS/25WC sintered in a vacuum furnace

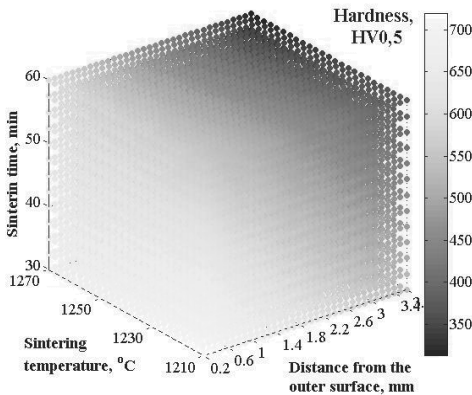


Figure 22. RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-90HSS/10WC, sintered in the furnace with the atmosphere of the flowing $N_2 + 5\% H_2$

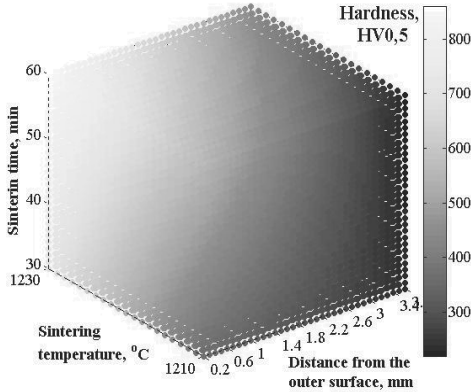


Figure 23. RFP describing relationship between hardness HV and the distance from the outer surface, temperature, and sintering time, for GM-75HSS/25WC, sintered in the furnace with the atmosphere of the flowing $N_2+5\% H_2$

The regression function plot describing the relationship between porosity and the fraction volume of the reinforcing phase in the individual layers and sintering temperature is shown in Fig. 24. It was found that the area with higher porosity in the materials sintered at 1210°C is limited only to the surface layers and disappears almost completely along with the growing sintering temperature.

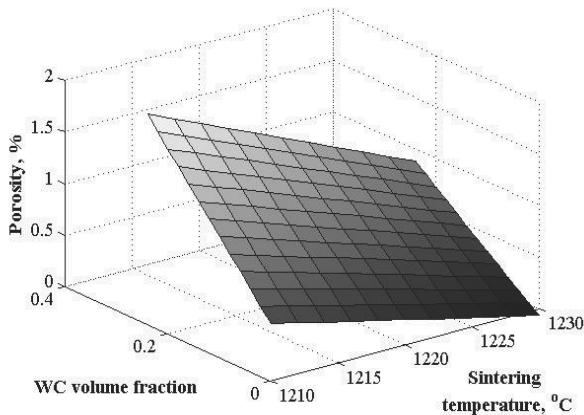


Figure 24. RFP describing relationship between porosity and the volume fraction of the reinforcing phase and sintering temperature, for GM-HSS/WC sintered in a vacuum furnace for 30 min

3.2. Sintering conditions and the reinforcing phase fraction versus GM-HSS/WC structure

It was found on the basis of metallographic research that such type of materials can be produced with a conventional method of powders metallurgy and both, the technological conditions of sintering and the fraction volume of the reinforcing phase impact the materials structure (Figs. 25, 26). The structure of the graded materials substrate layer is represented by alloy ferrite and MC and M_6C primary carbides (Fig. 27), whereas WC carbide and W_2C phase not occurring in the moulding exist in the intermediate layers and in the surface layer of graded materials (Fig. 28). Figs. 29 to 32 present the impact of sintering conditions on the structure of the substrate structure containing high-speed steel only, of intermediate layers and the surface layer of GM-90HSS/10WC and GM-75HSS/25WC.

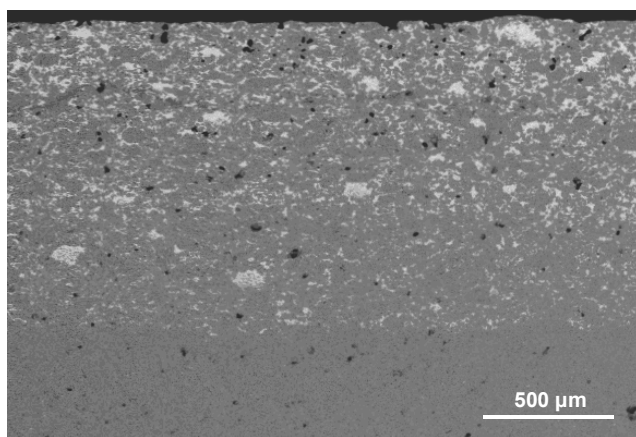


Figure 25. Microstructures of GM-90HSS/WC sintered in the vacuum furnace at the temperature of 1230°C, for 30 min

It was found by observing the structure of graded materials that the impact of temperature and sintering time is clear. The structure of the sintered GM-90HSS/10WC substrate layers has been changed considerably. Some of primary carbides are occupying the areas with concave surface and have an elongated shape characteristic for eutectic carbides. If sintering temperature is increased, the primary carbides will grow as a result in the material structure. A structure distinctive for remelted high-speed steel is present. Large carbides (approx. 5 μm)

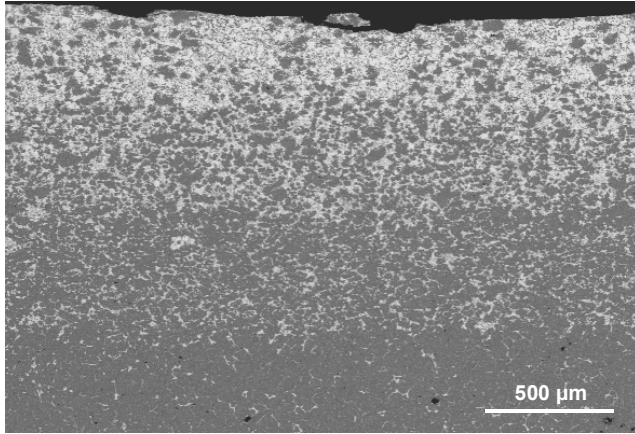


Figure 26. Microstructures of GM-90HSS/10WC sintered in the vacuum furnace at the temperature of 1230°C, for 30 min

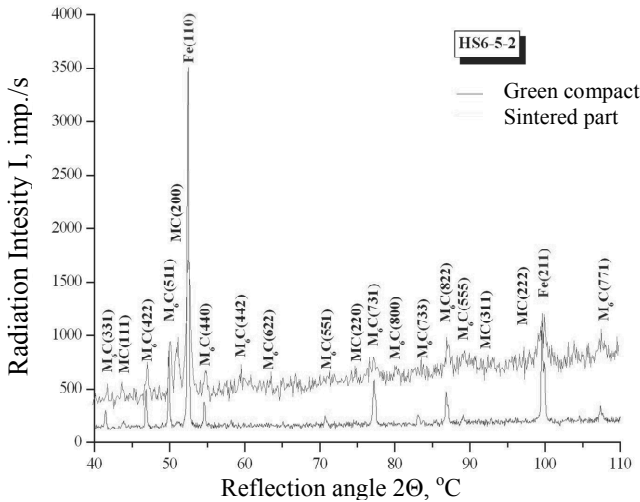


Figure 27. Results of the X-ray phase analysis for the sintered test piece with the HS6-5-2 layer; diffraction patterns were shifted along the vertical axis to show the results more clearly

are arranged mainly at the boundaries of grains and small carbides ($< 1 \mu\text{m}$) are arranged inside the grains. If the sintering temperature is raised, then the eutectic carbides in the substrate layer structure occupy larger and larger areas and, at the sintering temperature of 1270°C, fill spaces between the grains of high-speed steel. The growing volume fraction of the WC strengthening phase in carbide steels causes primary carbides to grow. The growth of primary carbides in the substrate layers structure of GM-75HSS/25WC can be explained with the fact that the carbon

originating from the dissolving WC carbide located in the intermediate layer and in the surface layer is reducing the sintering temperature. For this reason, while the fraction of WC carbide is growing, so is growing the concentration of the dissolved carbon thus reducing the sintering temperature value of the substrate layer (of high-speed steel) from 1250°C to 1210°C. A higher sintering temperature intensifies, most of all, proneness to the uncontrolled growth of carbides at the final stage of sintering.

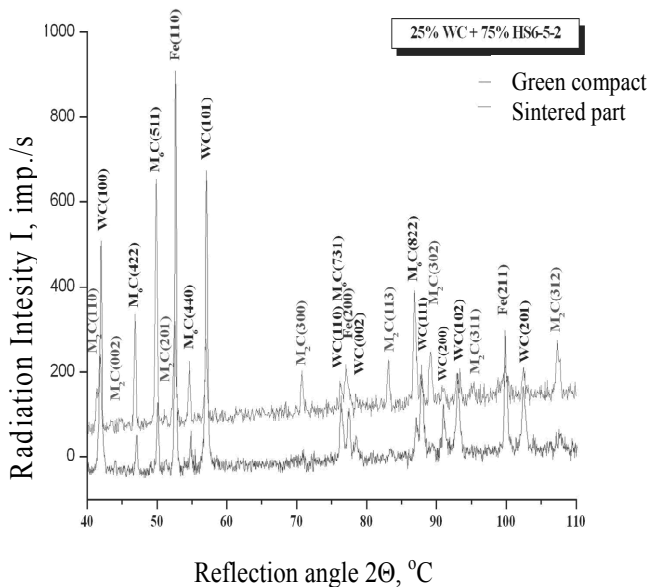


Figure 28. Results of the X-ray phase analysis for the sintered test piece with the 75HSS/25WC surface layer; diffraction patterns were shifted along the vertical axis to show the results more clearly

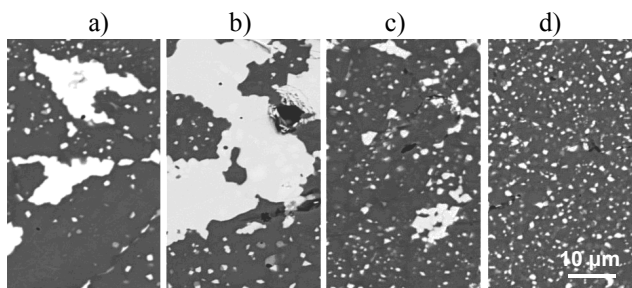


Figure 29. Layers structure of GM-90HSS/10WC sintered in a vacuum furnace at the temperature of 1210°C, for 30 min; a) surface layer, b), c) intermediate layers, d) substrate layer

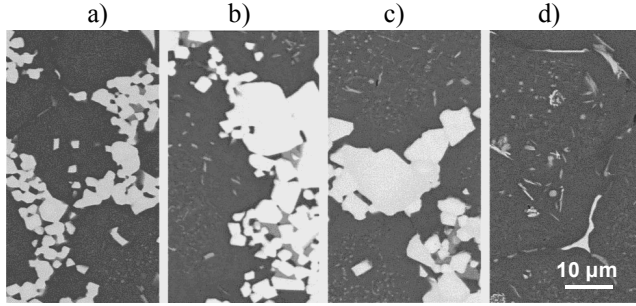


Figure 30. Layers structure of GM-90HSS/10WC sintered in a vacuum furnace at the temperature of 1270°C, for 60 min; a) surface layer, b),c) intermediate layers, d) substrate layer

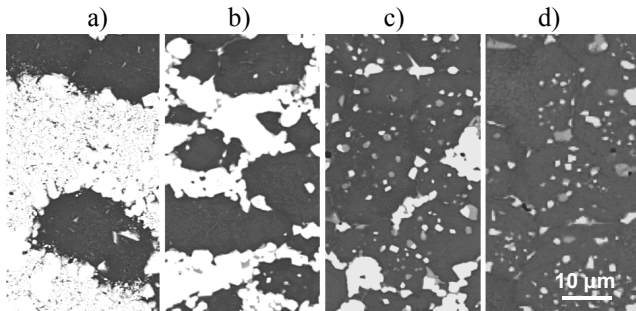


Figure 31. Layers structure of GM-75HSS/25WC sintered in a vacuum furnace at the temperature of 1210°C, for 30 min; a) surface layer, b),c) intermediate layers, d) substrate layer

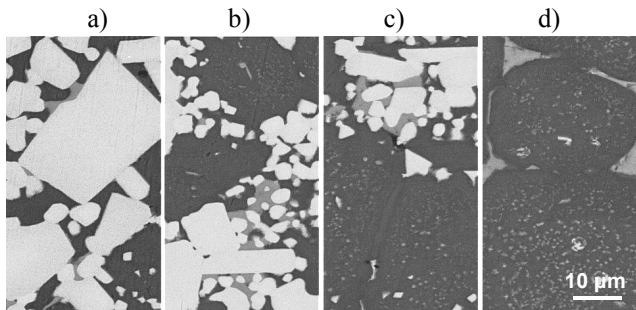


Figure 32. Layers structure of GM-75HSS/25WC sintered in a vacuum furnace at the temperature of 1230°C, for 60 min; a) surface layer, b),c) intermediate layers, d) substrate layer

3.3. Heat treatment conditions versus GM-75-HSS/25WC structure and properties

It was found based on the hardness tests that the impact of heat treatment conditions on the hardness of the tested graded materials in the quenched and tempered condition is noticeable in the substrate layers composed of the same high-speed steel (Figs. 33, 34). The HRC hardness tests of graded materials substrate layers in the heat-treated condition show a clear effect of the tempering temperature on the hardness value. The maximum secondary hardness effect of approx. 66.7 HRC was achieved in the materials austenitised at 1210°C for 80 s, quenched and tempered at 560°C. If the tempering temperature is raised to 590°C, hardness is lowering as compared to the condition corresponding to the secondary hardness effect, by approx. 1 HRC – for the materials austenitised at 1180 and 1210°C and by approx. 2 HRC – for the materials austenitised at 1150°C (Fig. 35). The greatest decline in hardness after tempering in the corresponding conditions, by approx. 3 HRC, has been seen for the substrate layers of the graded materials pre-quenched from the temperature of 1120°C.

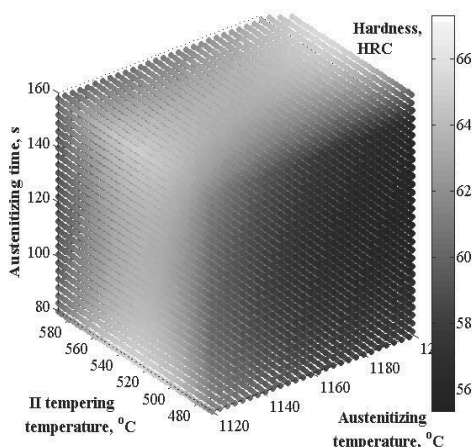


Figure 33. RFP describing relationship between hardness and the temperature, and austenitising time, and second tempering temperature for the GM-100HSS substrate layer of GM-75HSS/25WC

The hardness of the graded materials surface layer is within the range of 69.2-71.6 HRC (Figs. 36-38). Heat treatment performed in the conditions applied in work is substantially improving hardness in the surface layer of materials by 5.8-8.2 HRC up to the value of 69.2-

71.6 HRC. The material austenitised at the temperature of 1120°C for 120 s, quenched and then tempered twice at the temperature of 530°C shows the highest hardness of the surface layer of 71.6 HRC. No statistically significant change in the impact of heat treatment conditions within the investigated range has been found on the hardness of surface layers after quenching and tempering the graded materials containing 25% of WC.

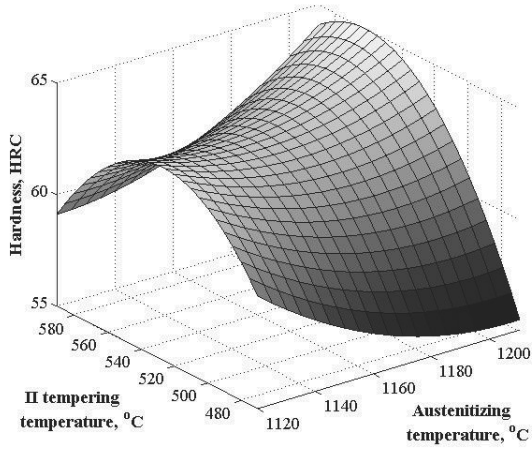


Figure 34. RFP describing relationship between hardness and the second tempering temperature, and the austenitising temperature, for the GM-100HSS substrate layer of GM-75HSS/25WC austenitised for 120 s

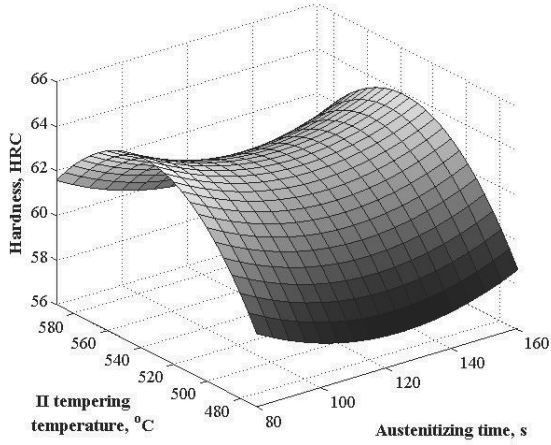


Figure 35. RFP describing relationship between hardness and the second tempering temperature, and the austenitising time, for the GM-100HSS substrate layer of GM-75HSS/25WC austenitised at the temperature of 1150°C

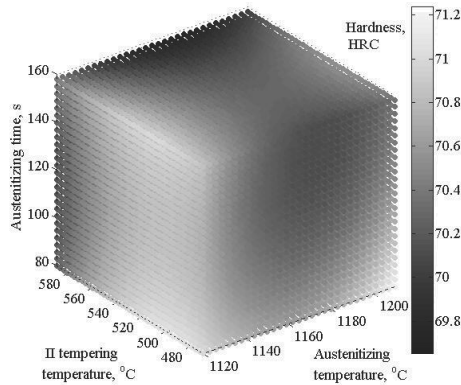


Figure 36. RFP describing relationship between hardness and the temperature, and austenitising time, and second tempering temperature for the GM-75HSS/25WC surface layer

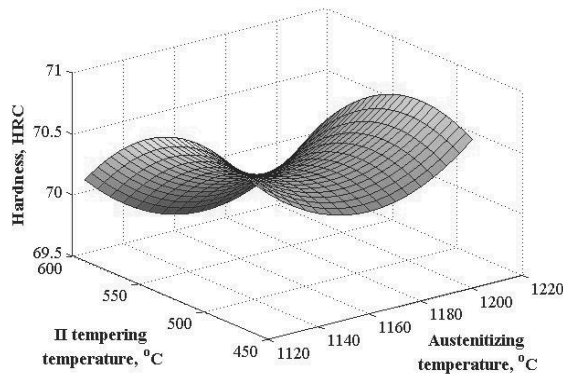


Figure 37. RFP describing relationship between hardness and the second tempering temperature, and the austenitising time, for the GM-75HSS/25WC surface layer austenitised for 120 s

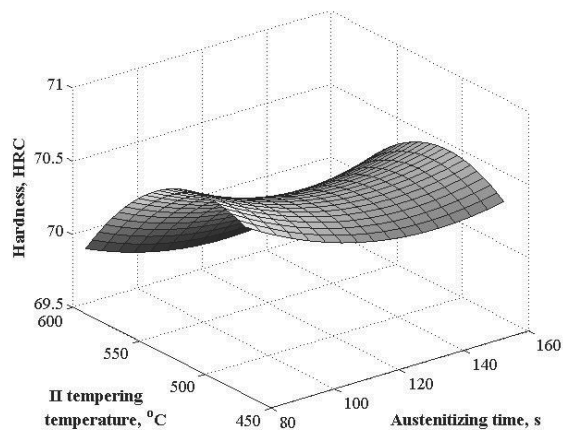


Figure 38. RFP describing relationship between hardness and the second tempering temperature, and austenitising time, for the GM-75HSS/25WC surface layer austenitised at the temperature of 1150°C

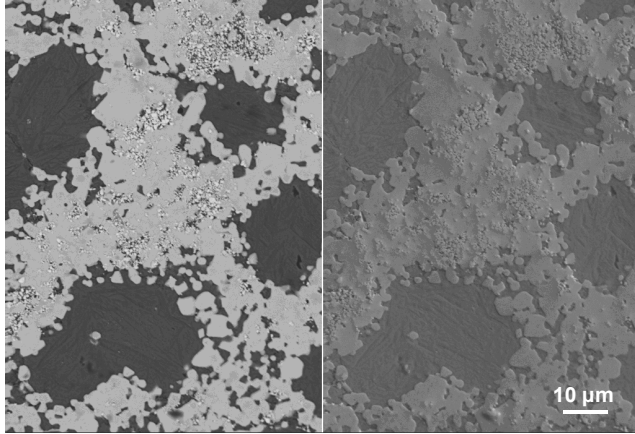


Figure 39. Structure of the quenched and twice tempered GM-75HSS/25WC surface layer

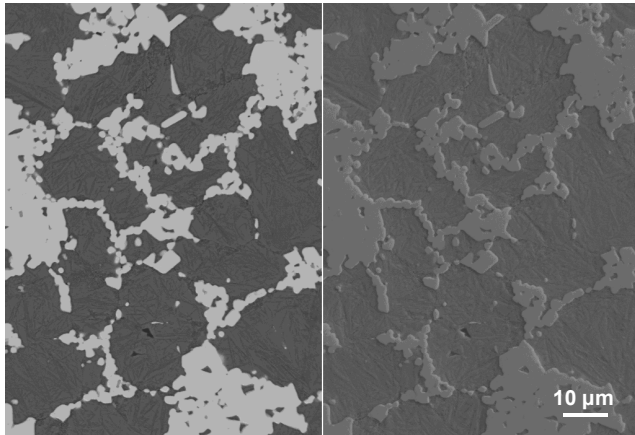


Figure 40. Structure of the quenched and twice tempered GM-85HSS/15WC intermediate layer

The structure of individual layers of the quenched and twice tempered GM-75HSS/25WC is presented in Figs. 39-42. The structure of the tested graded materials in the quenched condition represents martensite with residual austenite, M_6C and MC type carbides, both primary and secondary, unsolved in a solid solution during austenitising and WC carbides in the surface layer of materials (Fig. 43). The primary and secondary carbides unsolved in a solid solution during austenitising have a large effect on the grain size of the primary austenite. Fig. 44 presents the results of the analysis of the volume fraction of carbides calculated with quantitative metallography methods. Variations in the primary austenite grain size indicator acc. to Snyder-Graff in the substrate layers containing 100% of HS6-5-2 high-speed steel

of graded materials, depending on temperature and austenitising time are presented in Fig. 45. The substrate layer of the gradient carbide steel quenched from 1120°C after austenitising for 80 s shows the primary austenite grain of approx. 12 acc. Snyder-Graff. The primary austenite grain increases along with the higher austenitising temperature, reaching the indicator value of approx. 6 acc. to Snyder-Graff – after quenching from 1210°C. The extension of austenitising time also supports the growth of primary austenite grain, whereas the extension of austenitising time is less intensive than increasing the austenitising temperature.

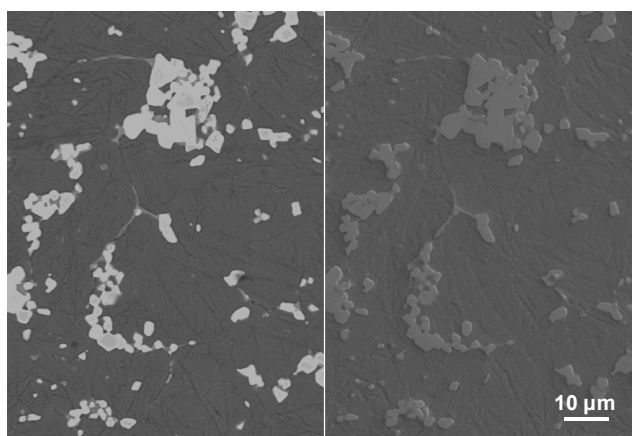


Figure 41. Structure of the quenched and twice tempered GM-95HSS/5WC intermediate layer

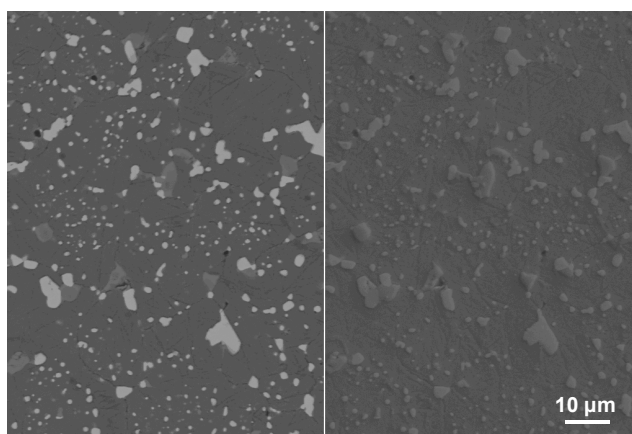


Figure 42. Structure of the quenched and twice tempered GM-100HSS substrate layer of GM-75HSS/25WC

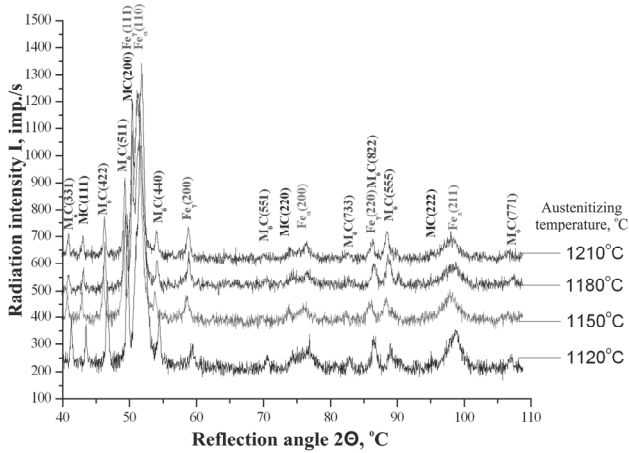


Figure 43. Results of the X-ray phase analysis from the GM-100HSS substrate layer of the GM-75HSS/25WC test piece, austenitised at the temperature of 1210°C, for 120 s; diffraction patterns were shifted in respect to the vertical axis to show the results more clearly

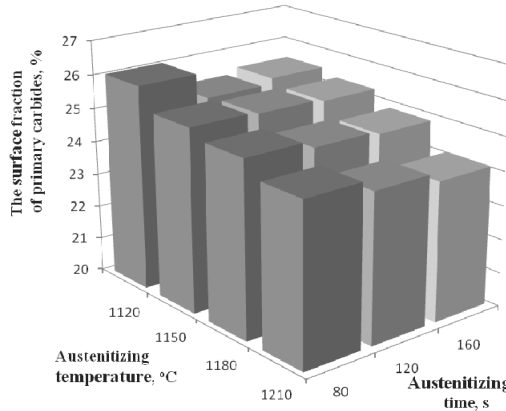


Figure 44. Relationship between the surface portion of carbides in the GM-100HSS substrate layer of hardened GM-75HSS/25WC and austenitising conditions

The matrix of the tested gradient carbide steels in the quenched condition from the temperature of 1210°C, ensuring maximum secondary hardness after tempering, is represented by martensite with residual austenite. The fraction volume of residual austenite in the structure of the tested substrate layer of the quenched gradient carbide steels is dependent upon the austenitising conditions. It was found with the X-ray quantitative phase analysis that the fraction volume of residual austenite of the quenched specimens is within the range of approx. 5.7 to 26.5% (Fig. 46). The fraction volume of residual austenite in the substrate layer of the gradient carbide

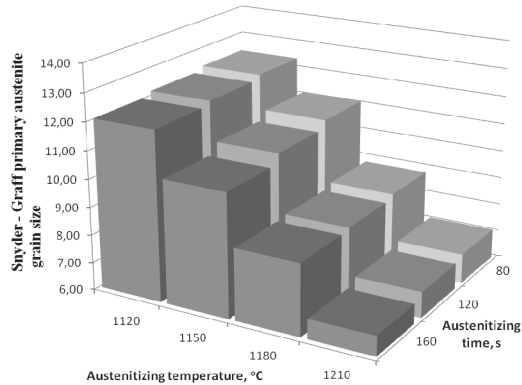


Figure 45. Relationship between the primary austenite grain size in the GM-100HSS substrate layer of hardened GM-75HSS/25WC and austenitising conditions

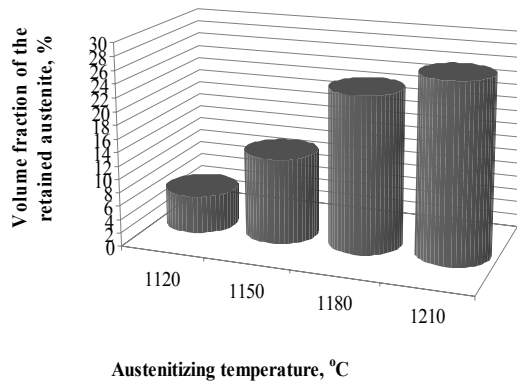


Figure 46. Relationship between the volume portion of the retained austenite and austenitising temperature in the GM-100HSS substrate layer of hardened GM-75HSS/25WC austenitised for 120 s

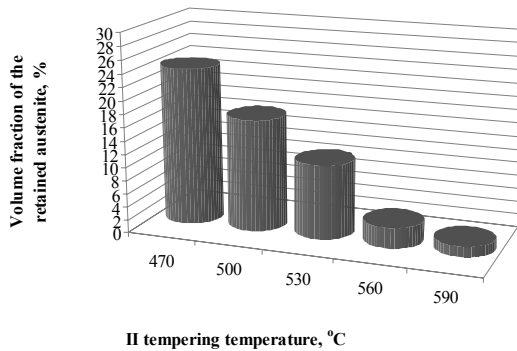


Figure 47. Relationship between the volume portion of the retained austenite with the second tempering temperature in the GM-100HSS substrate layer of GM-75HSS/25WC hardened at the temperature of 1210°C after austenitising for 120 s

steels quenched from the temperature of 1210°C, ensuring the maximum secondary hardness after subsequent double tempering within the temperature range of 470 to 590°C decreases and, according to the temperature of the second tempering, is within, accordingly, 1.6-23.8% (Fig. 47).

3.4. GM-3Co/97WC structure and properties

It was found based on the density measurements of sinters for the newly developed graded tool materials in the cobalt matrix that the material sintered using isostatic hot pressing and pressure sintering exhibits the highest density. The density of materials produced after sintering with isostatic hot pressing at the temperature of 1460-1425°C is, respectively, 14.60 g/cm³, and the density of the materials subjected to free sintering at the temperature of 1460°C, is 12.96 g/cm³, accordingly. It was found when analysing the impact of the sintering process parameters on density that the density increases while reducing porosity along with extending the process time and temperature. The X-ray quantitative analysis method performed using an EDS scattered radiation spectrometer (Figs. 48, 49) confirms that W, C, and Co element is present, respectively, in the hard phase of tungsten carbide and the binding phase of cobalt in the specific layers of the graded tool material. The newly developed graded tool material is characterised by a compact structure due to the uniformly distributed fraction of the binding phase between the hard carbide phase. It was confirmed through the tests of thin foils made in a transmission electron microscope (Fig. 50) that the sintered graded tool materials contain tungsten carbide and cobalt grains.

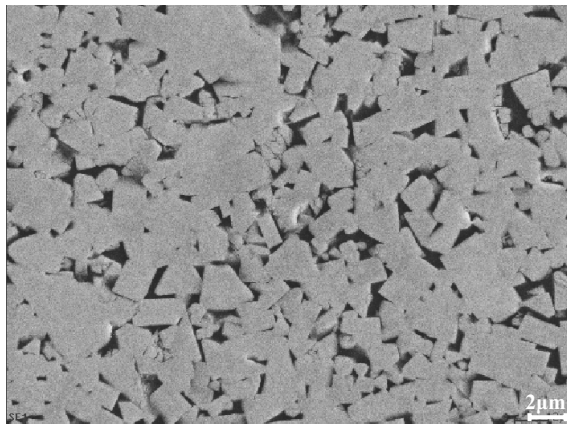


Figure 48. Structure of the GM-3Co/97WC surface layer sintered in a vacuum furnace at the temperature of 1460°C

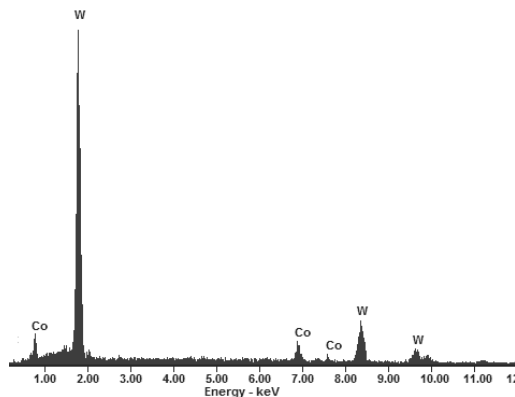


Figure 49. Diagram of intensity according to energy of scattered X-ray radiation for the whole area for the surface layer

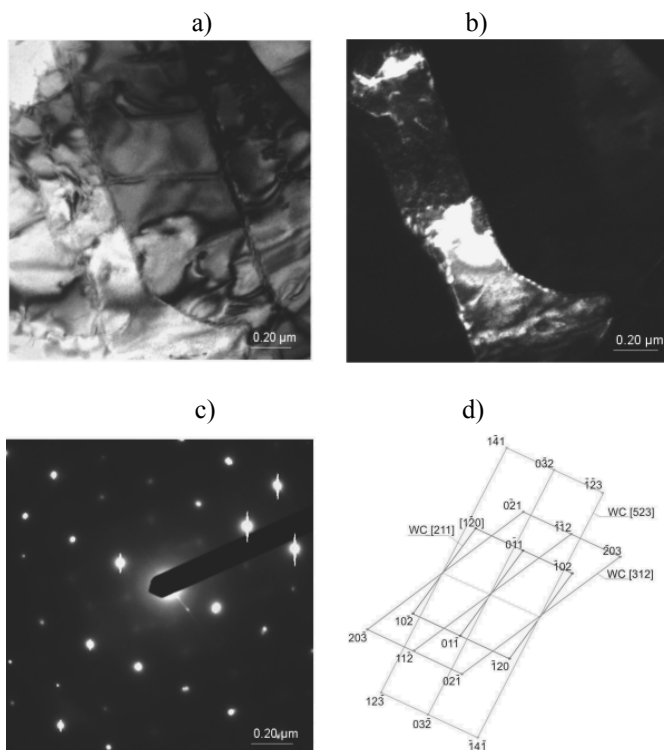


Figure 50. The structure of thin foils made of WC-Co sintered carbide after sintering at the temperature of 1460°C; a) image in the light field; b) image in the dark field of Co and WC; c) diffraction pattern from the area as in figure a; d) diffraction pattern solution from figure c [58]

The results of HV hardness measurement (Fig. 51) of the produced tool materials with the growing fraction of WC carbide in relation to the cobalt matrix towards the tool surface indicate gradual increase in hardness. The hardness of the material sintered at the temperature of 1460°C in vacuum is within 1410-1295 HV and decreases along with the growing distance of the measuring point from the outer surface of the surface layer to the substrate. The hardness of sintered material in 1460°C and pressed isostatically at the temperature of 1425°C is within the range of 1430-1326 HV in the surface layer and decreases towards the substrate.

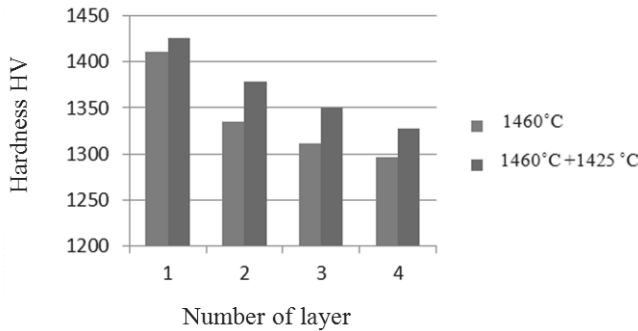


Figure 51. Diagram of HV hardness, fraction volume and sintering temperature for four layers of GM-3Co/97WC

The results of brittle cracking resistance tests K_{IC} for the sintered graded tool materials with a varied volume fraction of WC and Co phases in each layer of the material are presented in Fig. 52. The K_{IC} co-efficient results show there is a substantial relationship between sintering parameters and the cracking resistance of the individual tool materials [54, 59-61]. GM-3Co/97WC sintered at the temperature of 1460°C is characterised by high brittle cracking resistance. The average value of K_{IC} co-efficient of the material surface layer is $21 \text{ MNm}^{-3/2}$, and $16 \text{ MNm}^{-3/2}$ for the substrate. The fact that there is no clear difference for K_{IC} co-efficient in the surface layer and in the substrate of the materials sintered with isostatic pressing can be explained with the sintering time that is too long causing the gradient structure to fade partially or fully [27-28, 62]. The microscope observations of specimens fractures carried out (Fig. 53) are characterised by the systems of hollows and the convexities imparting the flaky character of the fracture, typical for brittle materials.

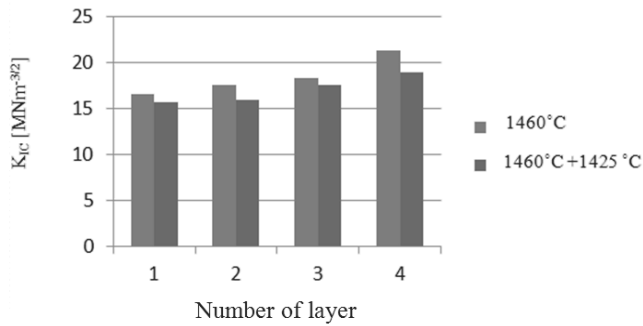


Figure 52. Brittle cracking co-efficient diagram according to temperature and fraction volume of Co for four layers of GM-3Co/97WC sintered in a vacuum furnace

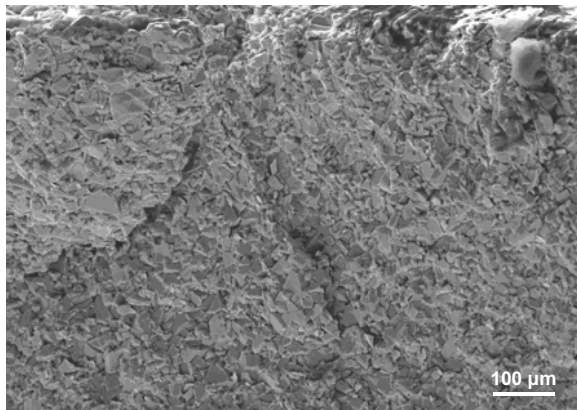


Figure 53. Fracture structure of the GM-3Co/97WC surface layer sintered in a vacuum furnace at the temperature of 1460°C

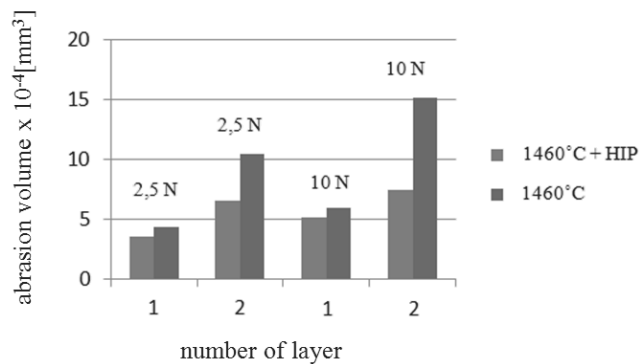


Figure 54. Tribological wear of investigated GM-3Co/97WC

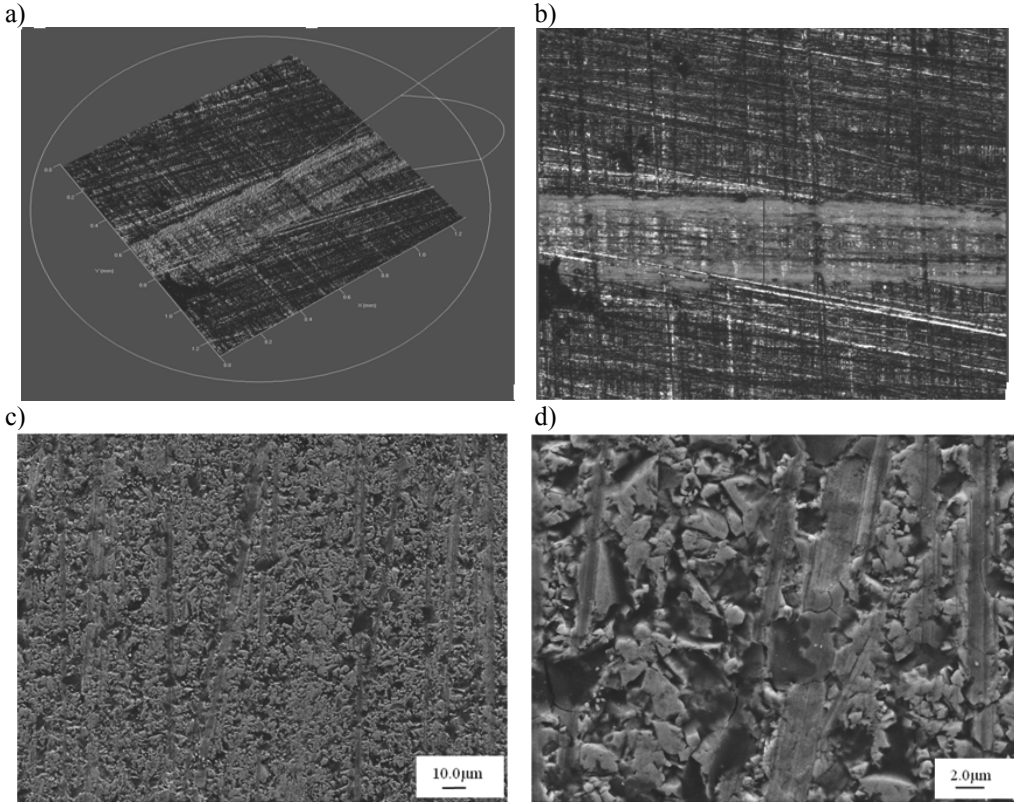


Figure 55. The wear track of GM-3Co/97WC sintered in vacuum in the temperature of 1460°C and pressed isostatically at the temperature of 1425°C after 1000 cycles with the load of 2.5 N in the substrate; a) CLSM micrograph; b) wear track measurement; c), d) SEM micrographs

The wear test results (Fig. 54) reveal that the materials sintered with isostatic pressing are characterised by much lower abrasive wear than those fabricated with free sintering. The material loss is caused by the detachment of particles due to micro-cutting or drawing in the counter-specimen – material friction area due to loose or restrained abrasant particles or the protruding irregularities of the hard carbide phase (Fig. 55) [63]. The unequal width of wear signifies the presence of seizing whereupon the wear products are being attached to the counter-specimens and then detached elsewhere causing local roughness where the wear is smaller. The presence of aluminium and oxygen, most probably originating from aluminium oxide Al_2O_3 has been confirmed with the X-ray quantitative microanalysis method carried out with an EDS scattered radiation spectrometer in the material wear track. This stems from the wear products being attached to the counter-specimens and then detached elsewhere causing local roughness where

the wear is smaller. The results of the abrasive wear measurement for the sintered graded tool materials of tungsten carbide in the cobalt matrix show the gradient shift of the tested materials properties depending on the presence of the binding phase. Numerous factors are, therefore, impacting the wear of graded materials: the presence of the binding phase, the counter-specimens load value and also the wear track (the number of cycles).

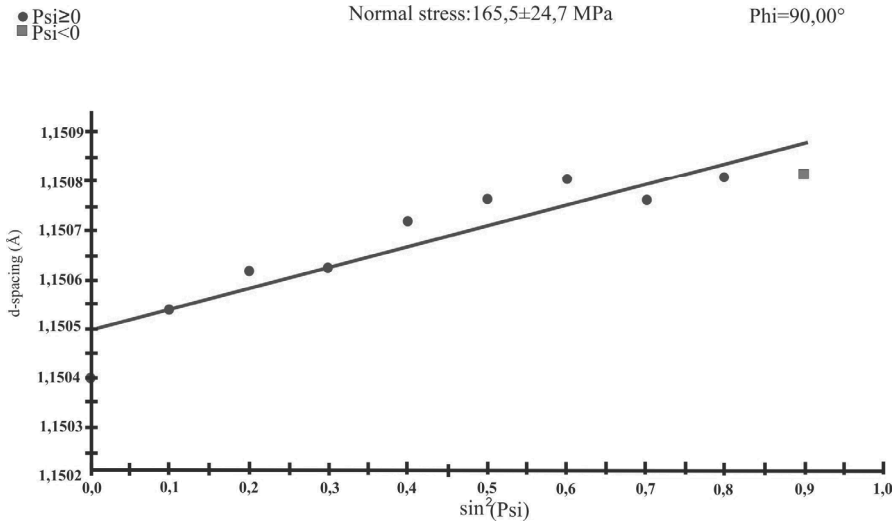


Figure 56. Changes in the interplanar distance value of d reflex (201) according to $\sin^2\psi$ sintering temperature of 1460°C; results for the GM-3Co/97WC surface layer

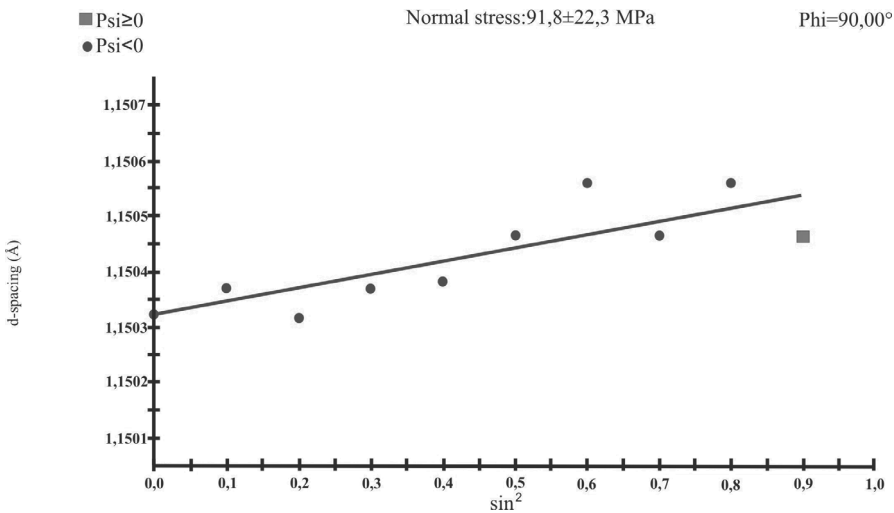


Figure 57. Changes in the interplanar distance value of d reflex (201) according to $\sin^2\psi$ sintering temperature of 1460°C; results for the GM-9Co/91WC substrate layer

The results of calculations of internal stresses in the tested materials obtained with an analysis of reflex shift (201) with the $\sin^2\psi$ method performed to verify the modelling results are presented in Figs. 56, 57 and in Table 4. The results obtained with a computer simulation of internal stresses with the finite element method match the results of the stress measurements obtained with the $\sin^2\psi$ method (Table 4).

Table 4. *The results of stresses obtained experimentally compared with the results of computer simulation*

	Sintering temperature, °C	Stresses determined experimentally, MPa	Simulated stresses, MPa
Surface layer	1460	162 ± 24	170
Substrate	1460	91 ± 22	80

4. Strategic position of graded tool materials manufacturing

4.1. Development perspectives of surface engineering of tool materials

The results of the technology foresight [6] performed with the sample size of 198 have revealed that tool materials surface engineering holds a stable position with respect to other thematic areas of surface engineering. According to 41% of respondents, tool materials surface engineering technologies are in the group of technologies with the extensive prospects of industrial applications. 32% of the respondents maintain that numerous scientific and research works will be frequently devoted in the nearest 20 years to the technologies. 35% of the surveyed claim that the thematic area of “Surface engineering of tool materials” is crucial and its importance should be absolutely rising so that an optimistic scenario can come true of the country's development – “Race won” – assuming that the potential available is adequately utilised to fulfil the strategic objectives of development and so that people, statistically, are better off, social attitudes are optimistic and the prospects for the coming years bright. 38% of the surveyed persons think that that the significance of tool materials surface engineering technologies in relation to other materials surface engineering technologies will be growing, whereas 61% assert it will remain on the same level with only 2 persons claiming that the importance will diminish over the next 20 years. In judging the anticipated role of tool materials surface engineering, its contemporary widespread industrial applications should be underlined and also

an ability to formulate freely the different, sometimes contradictory properties of the core and the surface layer of tools at a cost-effective level. Considering these reasons and the technology foresight results obtained, the future strategic position of tool materials surface engineering technologies should be considered stable and unthreatened, and their role in the development of overall materials surface engineering (mezo scale) and the development of the whole Polish economy (macro scale) will be certainly substantial.

4.2. Manufacturing of sintered graded tool materials in the future

The anticipated position of the powders metallurgy method allowing to change chemical and/or phase composition in the surface layer was determined based on the results of the own foresight research performed with the Delphic method [6]. The outcomes of the research indicate the strong development prospects of the materials that are still used mainly at a laboratory or semi-technical scale. It is also justified to apply them on a larger scale in the future in the industrial practice. 58% of the experts surveyed think that the technology group is critical and its importance should be absolutely rising so that an optimistic scenario of the country's development, i.e. "Race Won" comes true in the nearest 20 years.

The foresight-materials science research described herein include the assessment, made by the key experts, of the potential and attractiveness of the three specific technologies of manufacturing GM-90HSS/10WC, GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method against the environment. A ten-degree universal scale of relative states (1: min, 10: max) was used to assess the individual groups of technologies, and an action strategy for the specific technologies was developed based on it and the forecast strategic development tracks were devised.

The key experts valued the technologies with the ten-degree scale for their: business, economic, humane, natural and system attractiveness as well as for their creational, applicational, qualitative, developmental and technical potential. A weighted average for the criteria considered (attractiveness and potential) was calculated using a multi-criteria analysis, and a result obtained for the individual technologies was entered into the dendrological matrix of technologies value (Fig. 58). All the investigations conducted show that all the technologies analysed are in their embryonic phase (10) of development and are characterised by high

attractiveness and limited potential. The technologies were entered into the quarter of the dendrological matrix referred to as soaring cypress. The following technologies were evaluated very much the same: (B) and (C), respectively: (4.82, 8.11) and (4.51, 8.55). The (B) technology of manufacturing GM-75HSS/25WC ensures that the expected material properties are achieved including its density, porosity and surface layer hardness for sintering and for thermal treatment while maintaining the ductility of the high-speed steel substrate. The (C) technology of manufacturing GM-3Co/97WC permits to produce a material with a varying gradient of chemical composition in its individual layers. This relates to hardness in the individual material layers being gradually differed while securing expected resistance to abrasive wear, brittle cracking resistance and the values of tensile stresses in the surface layer ensuring resistance to the formation and propagation of cracks. The (A) technology of manufacturing GM-90HSS/10WC allows to achieve smaller hardness of the surface layer measured with the Vickers and Rockwell method as compared to the (C) technology.

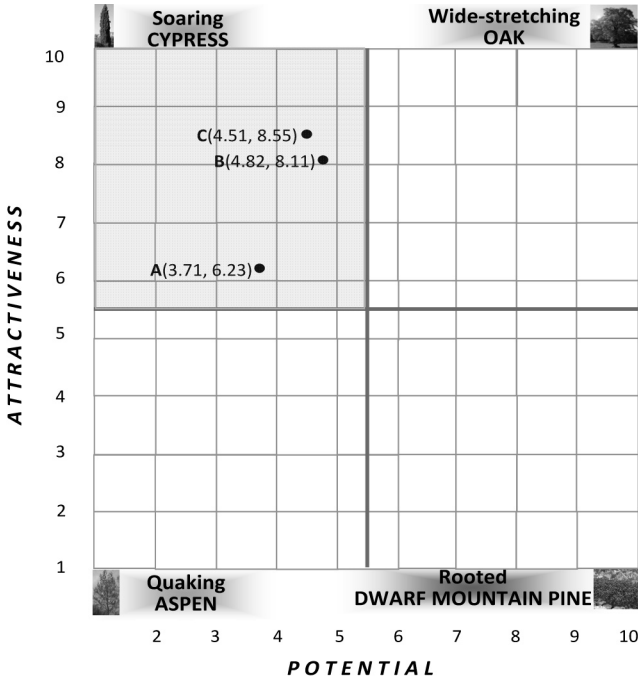


Figure 58. The dendrological matrix of technology value for the manufacturing technologies of the following sintered graded tool materials; (A) GM-90HSS/10WC, (B) GM-75HSS/25WC, (C) GM-3Co/97WC

Moreover, more time and a higher temperature for the (C) technology is required in the sintering process due to the lower contents of carbon in the powders mixture, hence it is less environmental friendly. For this reason the (A) technology has received relatively lowest scores from the experts (3.71, 6.23).

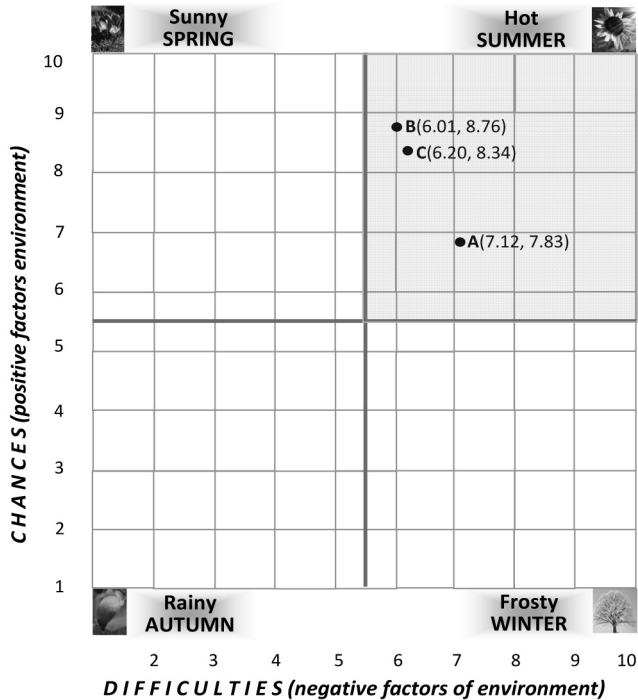


Figure 59. The meteorological matrix of environment influence for the manufacturing technologies of the following sintered graded tool materials; (A) GM-90HSS/10WC, (B) GM-75HSS/25WC, (C) GM-3Co/97WC

The evaluation results of the positive and negative environment influence on the relevant technologies were visualised with a meteorological matrix of environment influence, as illustrated in Fig. 59. The experts surveyed have found that the environment of all the technologies of manufacturing sintered gradient tool materials analysed experimentally is stormy. They were placed in the quarter of the meteorological matrix called hot summer for this reason. The environment of the technologies analysed brings ample opportunities related to the attractive prospect areas of future applications in the tool industry accompanied, however, by numerous difficulties such as fierce global competition and an alternative search for the effective technologies of manufacturing graded tool materials. Graded tool materials can also

be manufactured through laser treatment (remelting and alloying) and in Physical Vapour Deposition (PVD), notably in combination with the glow thermochemical treatment hybridised in this process [41]. The (B) group of technologies (6.01, 8.76) and the (C) group of technologies saw similar results (6.20, 8.34), and the (A) group of technologies had a weaker position with the result of (7.12, 7.83) which stands for fewer opportunities and more difficulties in the future.

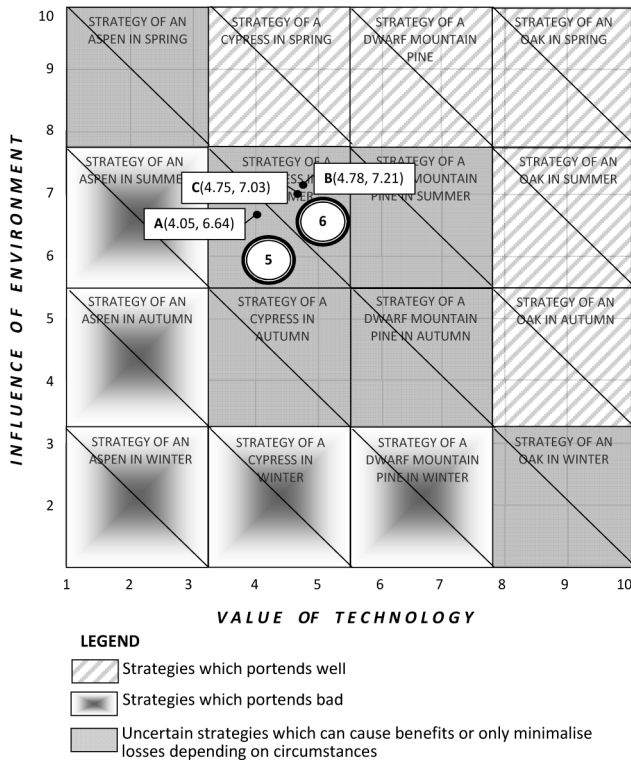


Figure 60. The matrix of strategies for technologies prepared for the manufacturing technologies of the following sintered graded tool materials: (A) GM-90HSS/10WC, (B) GM-75HSS/25WC, (C) GM-3Co/97WC

Using the pre-defined mathematical relationships, the specific numerical values provided in the following four-field matrices: the dendrological and meteorological matrix, were moved to the sixteen-field matrix of strategies for technologies (Fig. 60). The matrix depicts graphically the place of the examined technologies of manufacturing sintered graded tool materials according to their value and intensiveness of environment influence, indicating the relevant management strategy. The circles mark the strategic development prospects of a given group of technologies

expressed in numbers. It is recommended to apply the cypress in summer strategy with reference to all the technologies analysed. According to the experts' assessment, the development prospects of the (B) and (C) technologies are moderate (6 points), and medium for the (A) technology (5 points). The recommended cypress in summer strategy assumes that the potential of attractive technologies must be enhanced and further scientific and research works need to be pursued to establish the optimum parameters and conditions of the manufacturing process in the risky environment conditions, and also a risk assessment is required. Either a customer should be fought for aggressively or the technology should be phased out slowly from the market depending on the result of the assessment. The cypress in summer strategy recommended for the analysed experimental technologies of manufacturing the sintered graded tool materials belongs to the unreliable ones. The greatest advantage of the analysed, newly developed technologies is their attractiveness connected with the promising mechanical and functional properties of the so produced tools. The risk factors, on the other hand, are as follows: the scale of future scientific studies in this scope, availability of financial resources for such research and the pace of development and the effectiveness of alternative technologies of manufacturing graded tool materials in laser treatment, Physical Vapour Deposition and hybrid (PVD and thermochemical treatment) processes.

The forecast strategic development tracks for the individual technology groups were prepared based on the expert opinions in the next part of the research works according to the three variants: optimistic, most probable and pessimistic ones for the relevant time intervals of: 2015, 2020, 2025 and 2030. The (B) and (C) technologies, i.e. manufacturing, respectively, GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method are characterised by a moderate growth outlook evaluated by the experts at 6 points in a ten-degree scale. This evaluation is influenced by both, internal factors including further necessary scientific and research efforts to strengthen the potential of such technologies as well as external factors such as the sharply developing alternative technologies of manufacturing graded tool materials.

The most probable track of (B) technology development assumes that its potential is to be strengthened in 2015-2020 through continued scientific and research works concerned with the formation of the hardness of the surface layer of graded carbide steels reinforced with WC depending on their chemical composition and sintering conditions. The research held up till now has revealed that GM-75HSS/25WC after sintering at 1210°C for 30 minutes exhibits the most promising mechanical properties. Heat treatment improves the hardness of this material's surface

layer considerably. The materials austenitised at 1120°C, quenched and then tempered twice at 530°C exhibit the highest hardness of the surface layer of 71.6 HRC. The adverse phenomena accompanying the (B) technology is that the specimen shrinks largely due to a high fraction of WC powder in the individual layers of the specimen subject to sintering related to the spatial deformation of the specimen. This disadvantage should be eliminated by reducing the fraction of tungsten carbide in the powders mixture and this requires further studies. The (B) technology will transit to the oak in summer field by strengthening the technology's potential in the years to follow (2025-2030) with the strong competition of alternative technologies. An optimistic track of the (B) technology development envisages extensive wide-scale research to strengthen the potential of the technology. As a consequence, the technology will be found in the oak in summer field already in 2020. The results of the research will allow to reinforce the position of the (B) technology versus the alternative technologies of manufacturing graded materials. This will eliminate in 2025 any major difficulties stemming from the environment and will allow for numerous industrial applications for the newly developed graded carbide steels reinforced with tungsten carbide and with the corresponding transition to the best field of the matrix called oak in spring. A pessimistic variant of (B) technology development, which is also probable due to its embryonic phase of development, envisages an insufficient interest from scientific and research and industrial circles. Its attractiveness will be declining gradually in 2015-2020 as a result with its limited potential being maintained and transition to the cypress in winter (2020) will be seen. The phenomena will be accompanied by the expansion of alternative technologies of manufacturing graded materials in laser treatment, Physical Vapour Deposition and (PVD) and hybrid (PVD and thermochemical treatment) processes causing further degradation to the (B) technology in the years to come (2025) and transition to the aspen in winter field with being eliminated entirely from the market.

The forecast development tracks of the (C) technology: most probable, optimistic and pessimistic are very similar to those predicted for the (B) technology. The future development of manufacturing GM-3Co/97WC with the conventional powder metallurgy method will be dependent upon the scale of scientific and research work conducted in this regard and the pace at which the alternative technologies of manufacturing graded tool materials are evolving. The directions of future research should focus on determining the optimum proportions of the cobalt and tungsten carbide powder mixture and on optimising the manufacturing process conditions including the crushing of powder mixture, forming the moulded pieces and sintering.

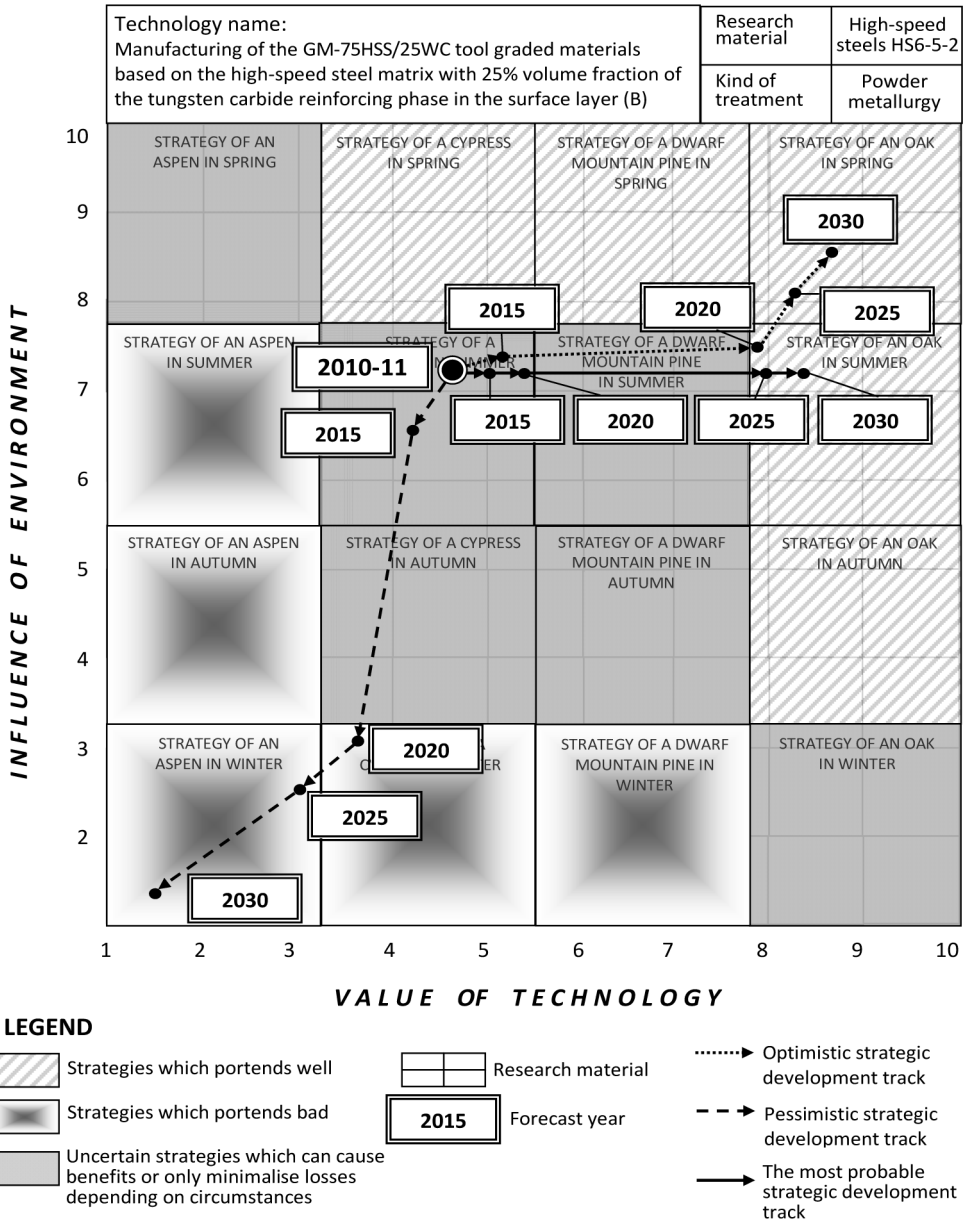


Figure 61. Exemplary strategic development tracks prepared for the (B) technology:
Manufacturing of the GM-75HSS/25WC sintered graded tool material

This is because these factors are decisive for hardness, wear resistance, resistance to brittle cracking and the values of tensile stresses in the material surface layer, being crucial for its strength to the formation and propagation of cracks. It should also be noted when conducting

future experiments that phase composition is equalising locally on the joint zones in the material that is heated too long (90 min) at a high sintering temperature of 1460°C and then compacted isostatically at 1425°C and the graded structure in the whole sinter volume is decaying.

Table 5. The strategic development tracks of sintered graded tool materials. Types of strategic development tracks: (O) – optimistic, (P) – pessimistic, (MP) – the most probable

Technology symbol	Technology name	Steady state 2010-11	Type of strategic development tracks	Years			
				2015	2020	2025	2030
(A)	Manufacturing of the GM-90HSS/10WC sintered graded tool material	Strategy of a cypress in summer A (4.1, 6.6)	(O)	(4.8, 6.6)	(7.9, 6.6)	(8.2, 6.9)	(8.6, 7.2)
			(P)	(3.1, 2.8)	(2.3, 1.8)	(1.0, 1.0)	(1.0, 1.0)
			(MP)	(3.7, 3.2)	(3.1, 2.8)	(1.7, 1.4)	(1.0, 1.0)
(B)	Manufacturing of the GM-75HSS/25WC sintered graded tool material	Strategy of a cypress in summer B (4.8, 7.2)	(O)	(5.2, 7.3)	(7.9, 7.4)	(8.3, 8.1)	(8.7, 8.6)
			(P)	(4.2, 6.6)	(3.7, 3.1)	(3.1, 3.6)	(1.5, 1.3)
			(MP)	(5.1, 7.2)	(5.4, 7.2)	(7.9, 7.2)	(8.3, 7.2)
(C)	Manufacturing of the GM-3Co/97WC sintered graded tool material	Strategy of a cypress in summer C (4.8, 7.0)	(O)	(5.1, 7.1)	(7.8, 7.3)	(8.2, 8.0)	(8.6, 8.6)
			(P)	(4.1, 6.5)	(3.6, 3.0)	(3.0, 3.4)	(1.4, 1.2)
			(MP)	(5.0, 7.1)	(5.3, 7.1)	(7.7, 7.1)	(8.2, 7.1)

The most probable development variant of the (A) technology, i.e. manufacturing GM-90HSS/10WC with the conventional powder metallurgy method that consumes lots of energy and time as compared to the (C) technology and produces the lower hardness of the surface layer of the manufactured material and worse, other mechanical and functional properties of tools in relation to the (C) technology [26,36,62], assumes that the technology, having its potential limited and its attractiveness diminishing and being forced out by other, more effective technologies, will move in short term (2015) to the cypress in winter field, and will then have been found in 2020 in the weakest field of the matrix of strategies for technologies, i.e. aspen in winter and will be eliminated from the market. The (A) technology development variant, which is unlikely, provides that scientific and research works will be undertaken due to certain properties of the tool produced (e.g. much smaller specimen shrink than for the (C) technology) aimed at strengthening its potential. As a result, the technology will move in 2020 to the oak in summer field and will stay there for the next years of the forecast. A pessimistic

(A) technology development track provides that its attractiveness will fall dramatically with a small potential and will degrade even faster than in the most likely case and transition will be seen to the aspen in winter field already in 2015 and rapid withdrawal from the market.

A graphical example of the (B) technology strategy matrix: of manufacturing GM-75HSS/25WC with the conventional powder metallurgy method with the strategic development tracks applied is presented for the three variants in Fig. 61. The numerical values, being the result of all the investigations pursued for the three analysed groups of technologies, are given in Table 5.

5. Technology Roadmapping

The results of the foresight–materials science research conducted represent reference data serving to create **technology roadmaps** being a comparative analysis tool enabling to choose a technology or a group of technologies which is best for the criterion selected. The roadmaps prepared with a custom concept have their set-up corresponding to the first quarter of the Cartesian system of coordinates. The following time intervals, respectively: current situation (2010-11), goals fulfilment methods (2020) and long-term objectives (2030) are provided on the axis of abscissa. Seven main layers ordered by their hierarchy are provided onto the axis of coordinates of the technology roadmap: time layer, concept layer, product layer, technology layer, spatial layer, staff layer and quantitative layer, made up of more detailed sub-layers. The upper-most layers of the technology roadmap are most general and determine the all-social and economic reasons and causes of the actions taken. The middle layers are characterising a product and its manufacturing technology. The bottom layers are determining organisational and technical matters concerning the place, contractor and costs. The cause and effect relationships, capital ties, time correlations and two-directional data and/or resources flow take place between the individual layers and sub-layers as signified graphically with the different types of arrows. Figure 62 presents a technology roadmap made for the (B) technology: manufacturing of GM-75HSS/25WC based on the high-speed steel matrix with 25% volume fraction of the WC reinforcing phase in the surface layer. Table 6 presents an aggregate list containing selected data being an extract from all the technology roadmaps developed under this chapter concerning sintered graded tool materials. **Technology information cards** are detailing out

and supplementing technology roadmaps. They contain technical information very helpful in implementing a specific technology in the industrial practice, especially in SMEs lacking the capital allowing to conduct own research in this field.

Table 6. Selected main source data used for preparation of technology roadmaps for investigated sintered graded tool materials

Analysed factors	Time interval	Manufacturing of the		
		(A) GM-90HSS/10WC	(B) GM-75HSS/25WC	(C) GM-3Co/97WC
		sintered graded tool materials		
Live cycle period	2010-11 years	Embryonic (10)	Embryonic (10)	Embryonic (10)
	2020 year	Embryonic (10)	Prototype (8)	Prototype (8)
	2030 year	Embryonic (10)	Early mature (6)	Early mature (6)
Machine park modernity	2010-11 years	High (8)	High (8)	Quite high (7)
	2020 year	High (8)	Very high (9)	High (8)
	2030 year	High (8)	Excellent (10)	Very high (9)
Quality and reliability	2010-11 years	Medium (5)	Medium (5)	Medium (5)
	2020 year	Quite low (4)	Quite high (7)	Moderate (6)
	2030 year	Low (3)	Very high (9)	Very high (9)
Proecology	2010-11 years	Medium (5)	Medium (5)	Low (3)
	2020 year	Medium (5)	Moderate (6)	Medium (5)
	2030 year	Medium (5)	Very high (9)	High (8)
Staff education level	2010-11 years	High (8)	High (8)	Very high (9)
	2020 year	Quite high (7)	Quite high (7)	High (8)
	2030 year	Quite high (7)	Medium (5)	Medium (5)
Capital requirements	2010-11 years	Very high (9)	Very high (9)	Very high (9)
	2020 year	High (8)	Quite high (7)	Quite high (7)
	2030 year	High (8)	Medium (5)	Medium (5)
Production size determining profitability in enterprise	2010-11 years	Quite high (7)	Quite high (7)	Very high (9)
	2020 year	Quite high (7)	Medium (5)	Quite high (7)
	2030 year	Quite high (7)	Quite low (4)	Medium (5)
Production size in the country	2010-11 years	Minimal (1)	Minimal (1)	Minimal (1)
	2020 year	Very low (2)	Low (3)	Low (3)
	2030 year	Very low (2)	Medium (5)	Medium (5)

Note: Research results are presented in universal scale of relative state, where: 1 is minimal and 10 is excellent level.

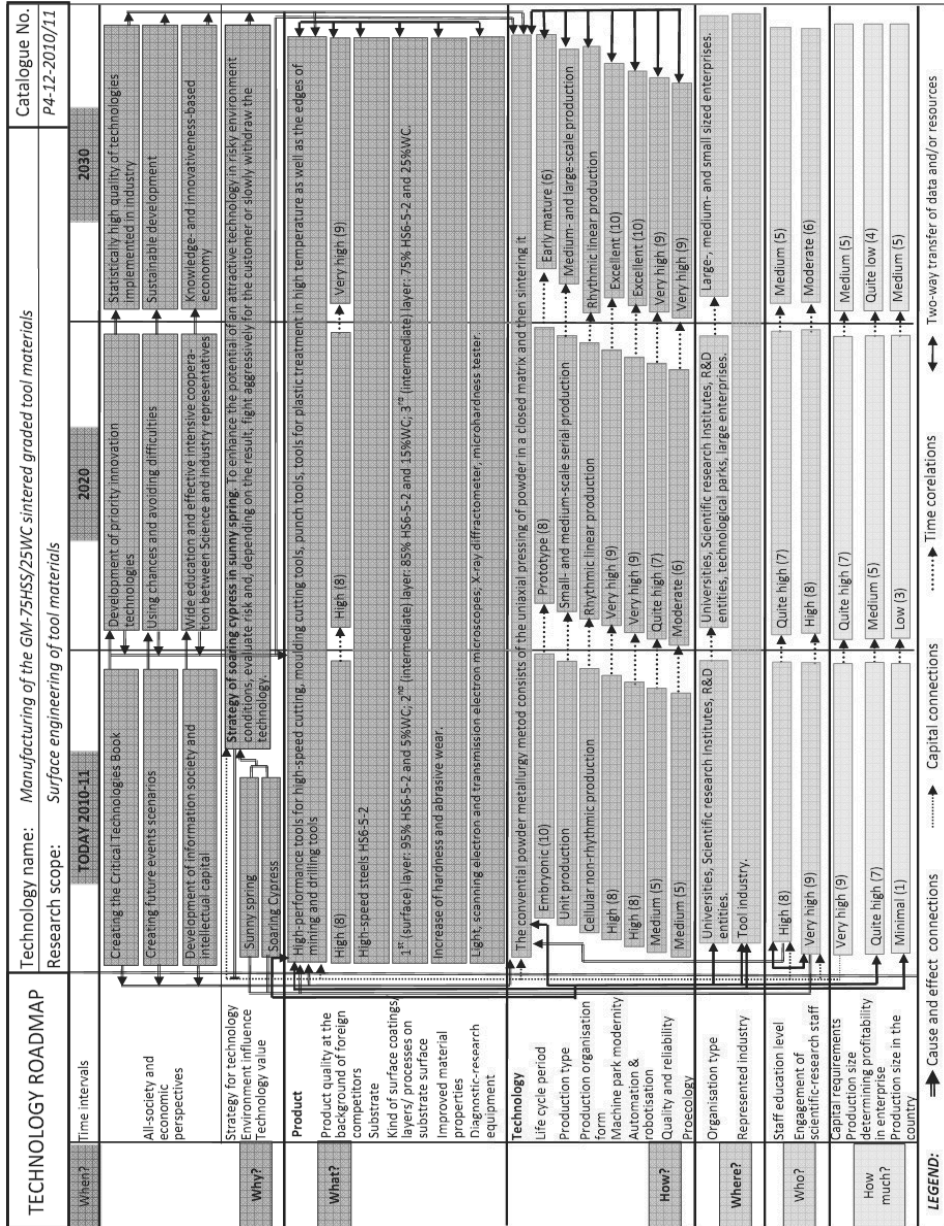


Figure 62. An example technology roadmap prepared for the (B) technology: *Manufacturing of the GM-75HSS/25WC sintered graded tool material*

6. Summary

This chapter presents the results of interdisciplinary experimental and comparative research concerning graded tool materials manufactured with the conventional powder metallurgy method.

Three specific technologies for manufacturing the following graded materials, i.e. GM-90HSS/10WC, GM-75HSS/25WC and GM-3Co/97WC are distinguished between for the purpose of the research followed by adopting the matrix material and fraction of powders in the mixture as a selection criterion. The materials science part of the work included in particular investigations into the influence of sintering conditions and the fraction of the reinforcing phase on the properties and structure of GM-HSS/WC, into the influence of heat treatment conditions on the properties and structure of GM-75HSS/25WC and into the structure and following properties of GM-3Co/97WC: density, hardness, toughness, abrasive wear, values of tensile stresses in the surface layer. A strategic position of tool materials surface engineering was identified for the foresight research in relation to surface engineering in general and a position of the powder metallurgy method, allowing to achieve changes to surface layer chemical composition and/or phase composition, in relation to other critical tool materials surface engineering technologies. The chapter also presents the results of the expert assessment of the potential and attractiveness in relation to the macro- and micro-environment of the three specific technologies of manufacturing GM-90HSS/10WC, GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method and action strategies for the technologies were formulated as well as forecast strategic development tracks determined. The results of the investigations are presented graphically using a set of matrices. Technology roadmaps were prepared at the final stage of the works illustrating, in a concise manner, basic information on the technologies analysed. The analysis made has revealed that the (B) and (C) technologies of manufacturing, respectively: GM-75HSS/25WC and GM-3Co/97WC with the conventional powder metallurgy method are characterised by moderate development prospects evaluated by the experts with 6 points in the ten-degree scale. According to the expert assessment, the development prospects of the (A) technology are medium (5 points). This severe score is affected by a high uncertainty with regard to internal factors including the necessity to continue scientific and research works to strengthen the potential of experimental technologies with high attractiveness as well as to external factors such as intensively evolving alternative technologies of manufacturing graded tool materials in the laser treatment, Physical Vapour Deposition and hybrid (PVD and thermochemical treatment) processes.

A surface layer can be provided with high resistance to abrasive wear while maintaining high core ductility at relatively low costs if the powder metallurgy method is applied. Such structure of the material allows to develop its properties freely depending on the working conditions of the tool. Hard surface layers can be used for example in the locations exposed to wear, and a ductile core can be used in other locations exposed to impact. It is predicted that

further scientific and research work should support the future wide industrial applications of the technologies analysed since the technologies have been used at a laboratory and semi-technical scale to date [64]. The advantages of manufacturing the sintered graded tool materials in industrial conditions include waste-free production and a short production cycle and a constraint is a possibility of manufacturing small-sized products with their shape determined with the construction parameters of dies. The specific properties of sintered graded tool materials make them suitable, notably, for use as high-performance tools for high-speed cutting, moulding cutting tools, punch tools, tools for plastic treatment in high temperature as well as the edges of mining and drilling tools.

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