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DENSIFICATION BY COLD RE-PRESSING OF LOW-CARBON MANGANESE STEELS

S. C. Mitchell, F. Baumgärtner

Abstract

Obtaining closed porosity, i.e. densities >7.4 g.cm^{-3}, is a major target in PM development. To increase density, strength and surface hardness of low-carbon PM steels: cold and warm compaction, sintering and slow cooling through the ferrite transformation region, followed by cold repressing and surface hardening were investigated. The slow cooling resulted in soft, ferritic, microstructure amenable to cold resizing. Repressing at 700-900 MPa densified the samples to ~7.6 g.cm^{-3}. Mechanical properties, after repressing and surface hardening, are characterised by appreciable plasticity following macroscopic yielding at stresses of 400-1200 MPa. Reference is made to possible further increases in strength by incorporation of small additions of clean, fine Mn containing master alloy into the powder mix. Results were verified industrially on hollow cylinders made from Fe-0.5Mo or Fe-1.5Cr-0.2Mo base powders.

Keywords: density, cold re-pressing, surface hardening, master alloy

INTRODUCTION

Use of ferrous PM parts can only be extended to high performance structural, i.e. high fatigue resistance, applications by the attainment of higher sintered densities [1]. It is held [2] that obtaining closed porosity, i.e. densities >7.4 g.cm^{-3}, is the next major step and warm compaction is playing a significant part in this development [3]. In this paper this technique has been used, as well as densification by cold resizing of samples possessing a soft largely ferritic structure, obtained by controlled cooling from the sintering temperature of the hypoeutectoid steels, see Fig.1, Fe-0.85/1.5Mo-1Cr-(0.1-0.3)C.

EXPERIMENTAL PROCEDURES

Laboratory warm compaction and cold sizing experiments

Initial Cr-containing mixes were prepared at the University of Bradford using either QMP's MSP 1.5Mo or Höganäs Astaloy 0.85Mo as base powders by admixing chromium carbide and graphite. To produce 0.5Mo content steels an admixture of Höganäs ASC 100.29 was made to the 0.85Mo base powder. For the warm compaction experiments lithium stearate was used as lubricant with pressing carried out at ~120ºC. Later laboratory work was carried out on pure iron and Fe-1.5Cr-0.2Mo base materials with clean gas atomised Mn containing master alloy additions (<20 μm). Industrial verification was performed by Schunk Sintermetalltechnik, GmbH on either Fe-0.5Mo or Fe-1.5Cr-0.2Mo with gas atomised Mn-containing master alloy, classified to <20 μm, admixed with the relevant base powder.

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The powders were mixed in a Turbula mixer for ~ 15 min. Uniaxial pressing of 15.3 mm^2 square specimens was performed at 600 or 680 MPa. Densities were determined by weighing and volume measurement. Sintering was carried out at 1280°C in a semi-closed container within a tube furnace employing an atmosphere of 75% nitrogen and 25% hydrogen, dried to a dew point of approximately –60ºC. For samples, which were not being repressed, the furnace cooling was at ~ 4°C/min.

As warm compaction of Fe-1.5Mo-1C- 0.8C resulted in a density increase of only 0.2 $g.cm^{-3}$, it was decided to add a further densification step: cold working of the alpha phase. Accordingly two hypo-eutectoid compositions, Fe-1Cr-1.5Mo-0.3C and Fe-1Cr-0.5Mo-0.15C, warm compacted at 680 MPa and sintered at 1280ºC were selected.

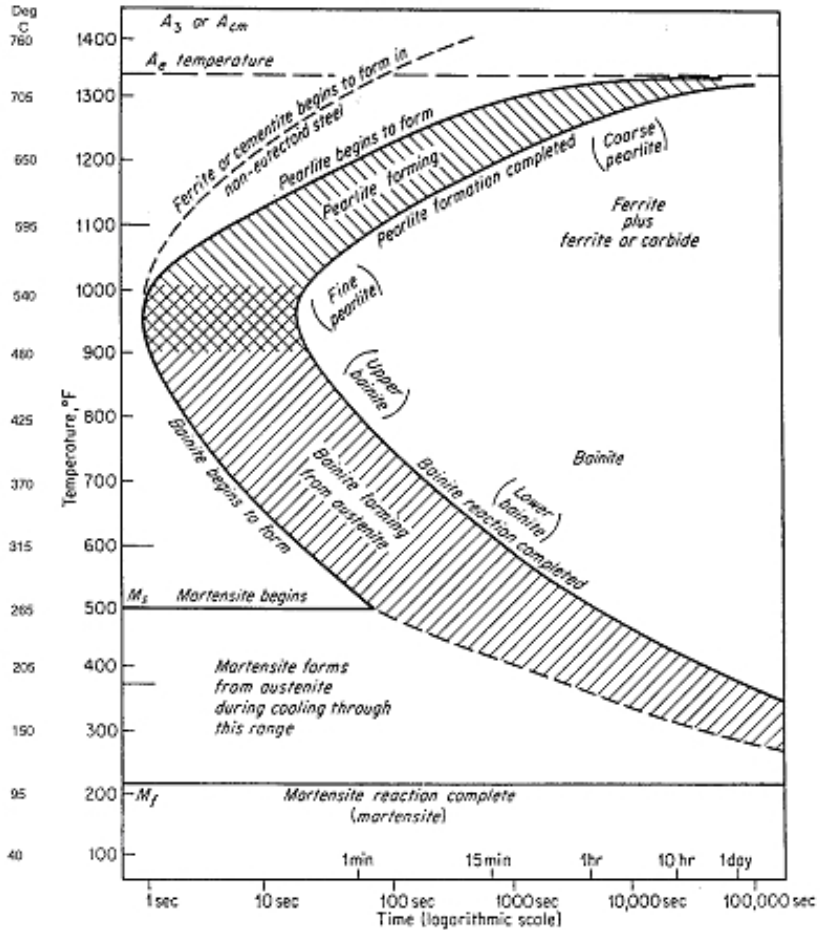


Fig.1. TTT curve for eutectoid steel. Please note the temperature range for the formation of pro-eutectoid ferrite.

To maximise ferrite formation, the samples were cooled at ~1ºC/min through the temperature interval 710-640ºC. Cold repressing, in the same die, was carried out at 700-900 MPa of specimens from the sides of which 0.1 mm was milled off. Table 1 lists the densities and bend yield strengths of the two alloys investigated. The specimens were then

either (a) Q&T: heated to 950°C in a vacuum furnace, gas quenched at 50°C/min and stress relieved at 200°C for 1 hour or underwent these surface treatments: (b) C, Q&T: cementation in a vacuum furnace in graphite powder at 950°C for one hour, quenched, reheating to 870°C for case refinement, quenching at 50°C/min and stress relieving at 200°C for 1 hour or (c) F, Q&T: flame hardening using proprietary "Casenit" powder followed by water quenching and a stress relief temper at 200°C for 1 hour. It is evident that for the 0.15% C material that the gas quenching rate was insufficient to provide adequate hardening. Optical and scanning electron microscopic examinations were carried out. Some specimens were slit into three prior to the hardening heat treatments and the beam samples then tested in three-point bending. Bend yield strengths, BYS, are presented in Tables 1 and 2.

Tab.1. Densities and bend yield strengths of the warm compacted alloys before and after sizing at 700, 800 and 900 MPa, respectively. [ND: not determined.]

Alloy	Sintered density [g.cm^{-3}]	BYS [MPa]	700 MPa sized		800 MPa sized		900 MPa sized	
			Density [g.cm^{-3}]	BYS [MPa]	Density [g.cm^{-3}]	BYS [MPa]	Density [g.cm^{-3}]	BYS [MPa]
Fe-1Cr-1.5Mo-0.3C	7.13	350	7.41	580	7.46	760	7.52	780
Fe-1Cr-0.5Mo-0.15C	7.22	260	7.50	ND	7.55	ND	7.58	ND

Tab.2. Heat/surface treatments and yield bend strengths, in MPa, of the alloys studied.

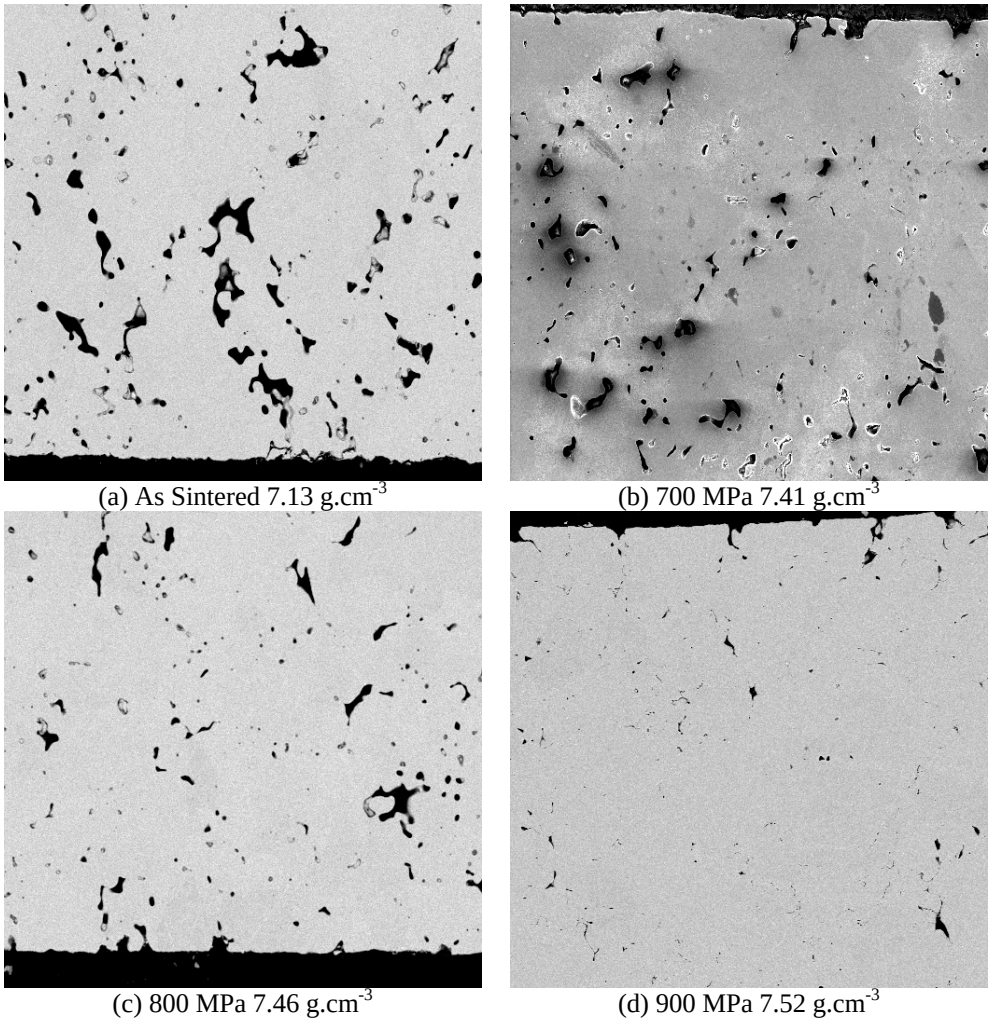
Alloy	No Sizing plus Surface treatment			Sizing at 700 MPa plus Surface treatment			Sizing at 800 MPa plus Surface treatment			Sizing at 900 MPa plus Surface treatment		
	Q&T	C,Q &T	F,Q &T	Q&T	C,Q &T	F,Q &T	Q&T	C,Q &T	F,Q &T	Q&T	C,Q &T	F,Q &T
Fe-1Cr-1.5Mo-0.3C	1100 MPa	N/A	N/A	1260 MPa	N/A	N/A	1280 MPa	N/A	N/A	1310 MPa	N/A	N/A
Fe-1Cr-0.5Mo-0.15C	320 MPa	380 MPa	420 MPa	430 MPa	540 MPa	1040 MPa	460 MPa	560 MPa	1180 MPa	480 MPa	590 MPa	1240 MPa

RESULTS

Laboratory Results

The densifications recorded in Table 1 result from the sizing operations alone. Consistently with the Fe-C phase diagram, there were ~80 and ~60% ferrite in the 0.15 and 0.3C alloys, respectively. The bend yield strength improvements seen in Table 1 were due to a combination of densification, work-hardening and limited cold welding of pores. In accord with the C contents, there was limited hardening in the 0.15C steel resulting from subsequent quenching and tempering (see column 1, Table 2), but increasing carbon to 0.3% resulted in very respectable bend yield strengths of ~1300 MPa. The flame hardening treatment (F, Q & T) was particularly effective with the re-pressed 0.15C alloy and bend yield strengths ~1200 MPa were achieved. The majority of specimens possessed

appreciable, >10%, ductility in bending, but no attempt at its quantification was made in these miniature specimens. Figure 2 shows typical porosity reduction with increasing resizing pressure.



(a) As Sintered 7.13 g.cm^{-3} (b) 700 MPa 7.41 g.cm^{-3}

(c) 800 MPa 7.46 g.cm^{-3} (d) 900 MPa 7.52 g.cm^{-3}

Fig 2. Reducing porosity with increasing repressing pressure.

Following the modest success of the previous experiments, mixes were made using either pure iron or Fe-1.5Cr-0.5Mo base powder to which was added clean Mn-containing master alloy powder and graphite. Compositions of the laboratory mixes were Fe-1.85Mn-0.1C and Fe-1.5Cr-0.2Mo-0.37Mn-0.15C. Specimens were pressed in the laboratory at 850 MPa, sintered in 95/5 N_2/H_2 atmosphere at 1280°C and slow cooled from 770-600°C to maximise ferrite content. Fe-1.5Cr-0.5Mo plus 0.2C was used for comparison purposes for green density, sintered density and as-sintered apparent hardness measurements. Sintering results are presented in Table 3. Repressing experiments were performed with various die clearances and good lubrication to discover the optimum

density, pressure and clearance combinations for each alloy. The results are presented graphically in Fig.3. To obtain potential core properties of the Mn-containing materials, tensile specimens were austenitised, oil quenched and stress relief tempered at 200°C. The ultimate tensile strength increased to more than 1.1 GPa with >1% plastic strain.

Tab.3. Density and slow-cooled hardness results for initial steels using atomised master alloy additions.

Sample	Composition	Green density [g.cm^{-3}]	Sintered density [g.cm^{-3}]	Density change [%]	Hardness HV5
39C/11	Fe-1.5Cr-0.2Mo +0.37Mn + 0.15C	7.24	7.34	1.32	98
39A/51	Fe + 1.85Mn + 0.1C	7.30	7.23	-0.90	106
Cr4	Fe-1.5Cr-0.2Mo +0.2C	7.27	7.31	0.65	109

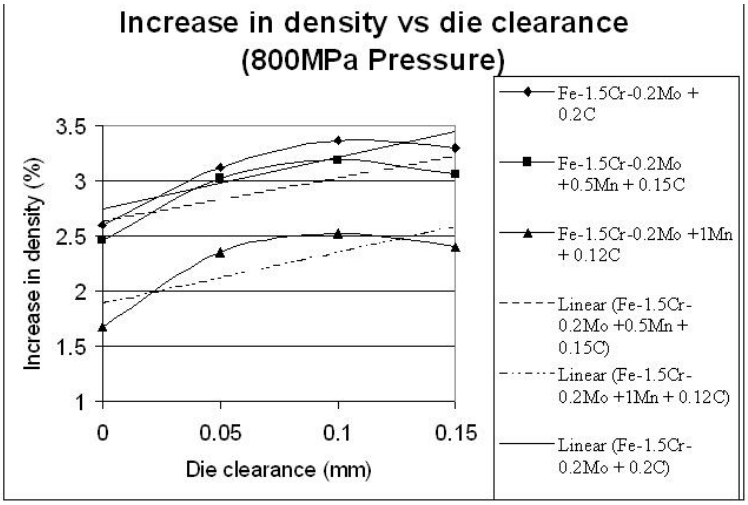


Fig.3. Typical coined density versus specimen die clearance results at constant re-pressing pressure in a well-lubricated die. Please note that increasing die clearance helps to promote material flow and thus improves densification (800 MPa pressure).

Industrial Results

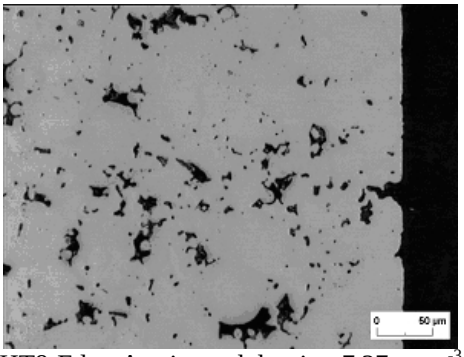
Following the success of the laboratory experiments, industrial trials were performed at Schunk Sintermetalltechnik, GmbH. Cylindrical samples, 19.9 mm OD x 8.1 mm ID, of composition Fe-1.5Cr-0.2Mo -0.5Mn-0.2C or Fe-0.5Mo-1Mn-0.2C were pressed at 700 MPa, sintered at 1180°C or 1250°C in 90/10 N_2/H_2 mixture, Fig.4. These specimens, whose apparent hardness varied from 100-120 HV10, were coined at 800 MPa and Table 4 lists the results. Typical reduction in porosity at both edge and core are shown in Fig.5.

Fig.4. Industrially pressed cylinders used for coining and surface hardening experiments. Outer diameter: 19.9 mm / Inner diameter: 8.1 mm.

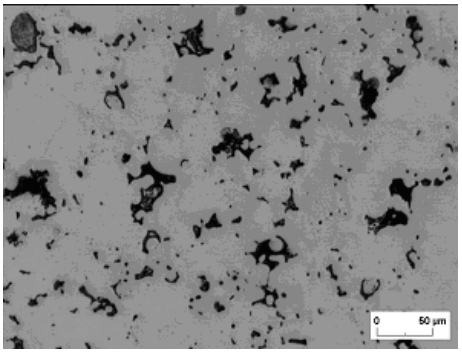
Tab.4. Green and sintered densities, as-sintered hardness and deviation from 19.9 mm OD-8.1 mm ID measurements.

HT8 1180°C	Sintered hardness HV10	OD	ID	Green density [g.cm^{-3}]	Sintered density [g.cm^{-3}]
Fe-1Mn-0.5Mo-0.2C	103	19.90	8.05	7.22	7.27
Fe-1.5Cr-0.5Mn-0.2Mo-0.2C	100	19.862	8.04	7.15	7.26
HT9 1250°C	Sintered hardness HV10	OD	ID	Green density [g.cm^{-3}]	Sintered density [g.cm^{-3}]
Fe-1Mn-0.5Mo-0.2C	120	19.91	8.06	7.22	7.26
Fe-1.5Cr-0.5Mn-0.2Mo-0.2C	125	19.89	8.05	7.15	7.24

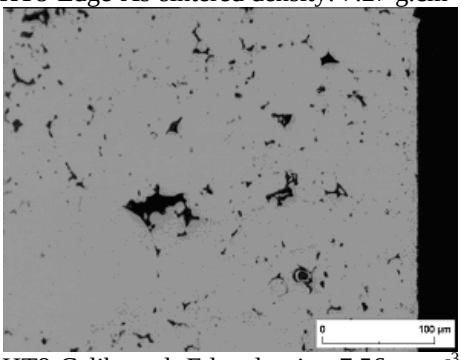
Cylinders were coined to both density and calibrate the samples to specified dimensional tolerance. Porosity distributions for both as-sintered and coined cylinders are shown in Fig.5. Carburisation and hardening experiments were performed and typical micrographs of edge and core are shown in Fig.6.



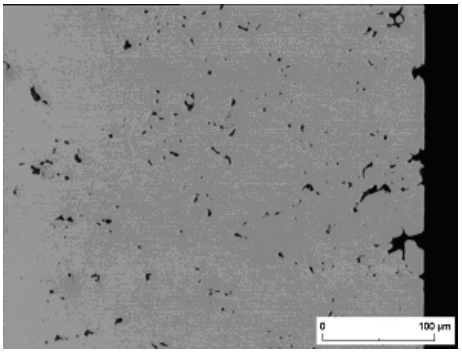
HT8-Edge-As-sintered density: 7.27 g.cm^{-3}



HT8-Core-As-sintered

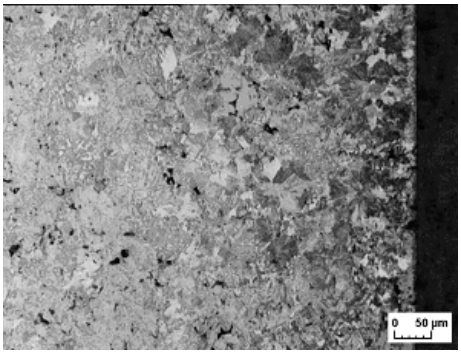


HT8-Calibrated -Edge density: 7.56 g.cm^{-3}

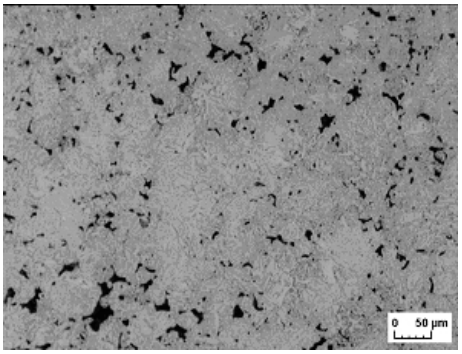


HT8-Calibrated-Core

Fig.5. Micrographs of Fe-0.5Mo-1Mn-0.2C cylinder material at edge (left) and core (right). Top as-sintered: 7.27 g.cm^{-3}, and bottom pair showing reduced porosity after coining to 7.56 g.cm^{-3}.



HT8-Calibrated -Carburised-Edge



HT8-Calibrated-Carburised-Core

Fig.6. Micrographs of carburised and quenched Fe-0.5Mo -1Mn-0.2C cylinder material at edge (left) and core (right) showing well-defined case hardened martensitic structure and bainitic core.

DISCUSSION

The use of re-pressing/sizing of a mainly ferritic microstructure following sintering seems an attractive potential production route. Reference should be made to the CCT curve of the relevant alloy; that for Fe-1.5Cr-1Mn-0.25Mo-0.2C is reproduced as Fig.7. Energy use is minimised by utilisation of the austenitising temperature during case

carburisation to perform a second sinter before hardening, thereby 'welding' some of the more flattened pores due to diffusion. The actual method of carburisation needs to be considered carefully in the light of available equipment. The principles are well–explained in the standard textbooks of e.g. Šalak, "Ferrous Powder Metallurgy" [5] and German, "Powder Metallurgy Science" [6]. German emphasises that repressing (Fig.9.1, p.343) decreases the size of the component and, more importantly, improves the uniformity of the dimensions. He shows that the height compressive strain must be at least twice the tensile radial strain to ensure densification in repressing. The accuracy of the dimensions obtainable by sizing decreases with increasing density and strength of the material and increases with the accuracy of the tool [should be at least one more grade accurate than required part accuracy] and rigidity of the press [5]. German further considers (Fig.9.2, p.344) the relationship of density to repressing pressure, showing how, for initial densities of 6.54 to 7.23 $g.cm^{-3}$, the resultant densities after repressing nearly converge to ~7.45 $g.cm^{-3}$ for a repressing pressure of ~1 GPa. Thus it is advantageous to have a relatively soft, relatively low-density sintered material if repressing is to be carried out. German (p. 323) considers powder forging to be a combination of densification and flow under uniaxial forces. This stress description applies equally to re-pressing when die wall constraint is important in determining the actual stress condition. The pore collapse at the centre of the component being re-pressed is similar to that encountered under hydrostatic pressure while near to the surface pores experience higher shear, possibly leading to shear closure (Fig.8.24, p.323). The results shown in Fig.3 illustrate that when clearance between the edges of the re-pressed compact and the die is >0.1 mm that densification begins to fall due to inadequate pressure distribution to produce pore collapse. As it is difficult to apply sizing and re-compacting to high-strength parts because of the high hardness [5], it is best to use a largely ferritic structure and "compensate" by a case hardening heat treatment.

This, again, has been considered by e.g. German [6] for vacuum carburisation in terms of relationships between hardness, and its extent below the surface, and porosity (Fig.9.9, p.351). He reports that the higher the porosity, the deeper the carbon penetration, because of permeation through the open pore network [by gaseous diffusion]. Below 8% porosity, the carburisation process is largely by (the far more sluggish) solid-state diffusion. As in atmosphere sintering it is again the gas-solid reactions that play the dominant role [7].

As to the specific system now reported on, further work needs to be performed on the actual alloy composition, which would probably benefit from addition of nickel and maintaining molybdenum content <0.5 wt%. This would ensure optimum energy utilisation during heat treatment (no core refinement necessary) and the best combination of core strength and toughness coupled with a high hardness case. Additional work would then need to be performed on resizing height change versus resizing pressure to obtain the necessary production data for initial green and final dimensions. The small amount of Mn introduced via the fine, clean atomised master alloy can only improve the core microstructure developed during heat treatment. A combination of strength and toughness produced by a well-developed bainitic core microstructure should serve well to produce a material whose fatigue strength is higher than that normally seen in PM components. These measures should ensure suitability for gear type applications [1,4].

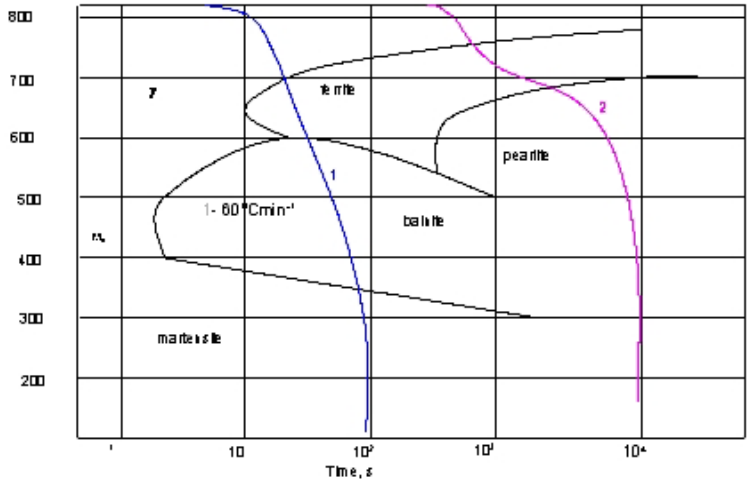


Fig.7. CCT Curve for Fe-1.5Cr-1Mn-0.25Mo-0.2C. Slow cooling profile 2 is used to promote ferrite-pearlite microstructure (Diagram courtesy Dr. A. Cias, AGH Krakow).

Acknowledgements

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