CURRENT STATE IN THE DEVELOPMENT OF PM TOOL STEELS, PROCESSING OF TOOLS AND PRACTICAL EXPERIENCES

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Abstract
The application of rapid solidification enabled us to produce steels of an excellent quality. The use of these materials leads to the significant cost saving due to the high stability of the production and minimisation of the risk of the sudden breakage of tools. Subsequent surface processing brings an additional progress in the tooling since these techniques can influence the surface quality in a desirable manner. The surfacing of tool steels usually covers the plasma nitriding or PVD – layering or, the duplex – coating as a combination of these two processes. The component of the duplex – layers work in a synergic principle whereas the nitried inter – layer increase the fatigue life – time, acts as a support for the PVD – layer, and this thin layer protects the tool against the wear. Practical experiences in cold work tooling confirmed that the complex of the substrate, made from fine grained P/M tool steel, processed via the duplex – coating, brought a considerable prolonging of the life time of tools and high production stability.

Keywords: powder metallurgy, ledeburitic steels, heat treatment, surface engineering, tooling, duplex-coating

INTRODUCTION
The history of high speed steels, and ledeburitic steels in general, goes back to the 1860 s, when the beneficial effect of tungsten on the hardenability and the heat resistance had been observed. However, the industrial use of tungsten was impossible due to its high price. The first successful industrial trial with alloying with tungsten is dated as recently as thirty years later and is connected with the establishment of the steel containing 1.85% C, 4% Cr and 7.5% W. In 1900, at the Paris World Exhibition, that material was firstly designated as the “High Speed Steel”.

The observation and enlargement of the chromium ledeburitic steels is connected with the lack of alloying elements available on the market, due to World War I, and requirements with respect to the labour productivity in France. The use of the Cr-steels for the cutting operations however, did not bring the expected effect because of an insufficient warm resistance. On the other hand, excellent wear resistance in the cold work applications was observed early and as a consequence, these steels became the most important group used for cold work applications, like sheet metal forming etc.

Since then, the scale of produced high alloy tool steels has been considerably broadened to the present-day range. Nowadays, several types of non-cobalt high speed steels, cobalt-containing materials, Cr-ledeburitic and sub-ledeburitic cold work steels and P/M materials are known and applied widespread, and the development in that material field is surely not yet finished.

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INGOT METALLURGY AND CONNECTED PROCESSES

Ledeburitic high alloyed tool steels have been produced by various types of casting procedures. In these processes, a slow solidification of a large volume of melt takes place, with a lot of undesirable consequences to the structure and properties [1].

The solidification usually begins with the formation of $\delta$-ferrite. The first crystals nucleate at the wall of the crucible and grow towards the centre in a dendritic manner. As the temperature decreases, solidification process continues by the thickening of dendrites. Simultaneously, residual melt is more and more enriched in carbon and alloying elements, up to a point where the formation of austenite is preferred instead of the $\delta$-ferrite. Besides the direct crystallization from a melt, the austenite may also appear in the structure as a result of peritectic transformation between the $\delta$-ferrite and the remaining melt as well as the eutectoidal decomposition of the $\delta$-ferrite ($\gamma +$ carbides).

In this stage of solidification, the residual melt has the maximal concentration of carbon and alloying elements and solidifies by the eutectic reaction. The eutectic reaction, however, is not as simple as that known from the Fe – C equilibrium diagram, but it contains several stages resulting in various eutectic mixtures. Their nature is, of course, depending on the actual cooling rate and chemical composition of the steel [2-5].

The solidification of the ledeburitic steels proceeds in a wide temperature range, usually more than 200 K. It makes serious problems for their metallurgical preparation because the multi-stage containing freezing mechanism leads to a large redistribution of alloying elements and to the formation of continuous carbide networks in particular. They make the as-cast steel brittle and inappropriate for direct use in most cases.

Hot rolling is then needed to break down these eutectics and make the steel more resistant to a brittle fracture [6]. Nevertheless, the carbide bands responsible for the anisotropy of mechanical properties originate as a result. Such a structural anisotropy causes the difference between the mechanical properties in longitudinal and transversal directions, with respect to the direction of carbide bands, which may make about 60% [7]. Structural inhomogeneity is finally reflected in the quality of manufactured tools.

The anisotropy of the mechanical properties is not invariable, but depends on many factors – mainly on the chemical composition. It increases as the alloying elements content increases. After that, the geometry of the initial as-cast ingot plays an important role – the larger the ingot the larger the segregation. Not least, of all an increasing deformation degree leads to a better homogenization of the microstructure and then to the lowering of the anisotropy.

Advanced melting techniques like the electroslag- and vacuum arc remelting (ESR, VAR) enable us to reduce the amount of impurities and segregation significantly. Typical symptoms for the as-cast ledeburitic steels such as the carbide networks however, appear also in the materials prepared in such a way, although their size and proportion is limited by improved freezing conditions. Moreover, the application of special remelting procedures increases the production costs, so that the price of the ESR- and VAR-steel exceeds that of the classic material several times over.

POWDER METALLURGY

As discussed above, for the ledeburitic steels it is difficult to obtain the as-cast and/or wrought structures with acceptable properties or to modify them in a sufficiently wide range [8]. Only the application of PM technique overcomes these problems and, in addition, extends metallurgical potentials for material production to a higher carbon and alloying elements content.
In most cases, the rapidly solidified (RS) powder, used as a starting semi-product in this fabrication technique, is obtained by gas atomisation. The use of water for the spraying process is no longer preferred; the surface contamination is too high and can make troubles during consolidation.

Because of the small dimensions of powder particles, typically between 10 and 300 μm, a rapid solidification effect takes place during freezing. The size of the RS powder as above can be referred to the cooling rate ranging between $10^3$ and $10^6$ Ks$^{-1}$ [9]. Such a wide range of freezing conditions, connected with the complicated crystallization mechanism described before, is reflected in the large scale of primary microstructures that is formed [10,11]. In addition, the following symptoms of non-equilibrium freezing are typical for powder production: reduction of grain or cell size, increased chemical homogeneity, extension of solid solubility. What is the most important fact for industrial application – the refinement of the structure is conserved also after consolidation of the RS powder.

The first attempts of the consolidation of RS powder were carried out via pressureless solid phase sintering. These experiments however, did not meet the expectations because a porosity of about 20 vol.% remained in the compact material. Liquid phase sintering overcame this problem [12]; unfortunately, the partial melting necessary for the achievement of full density makes a risk of the formation of continuous carbide networks, which deteriorates the mechanical properties rapidly. The use of hot isostatic pressing (HIP) brought together the requirements of both the full density and the avoidance of continuous carbide networks in the bulk products. Before encapsulation however, the powder has to be prepared and mixed carefully, in order to minimize unwanted shape distortion of the capsules. What has to be always taken into consideration is that dimensional changes take place during the consolidation as the porosity decreases? The temperature of HIP ranges usually between 50 and 100°C below the actual solidus temperature, the gas (argon in most part) pressure normally exceeds 100 MPa. As a result, the material has full density and is free of macro-segregation, see Fig.3. Afterwards, the bulk steel is submitted to soft annealing and to the removal of the capsule material from the surface. Prepared material by such a manner is then suitable for any machining operation and tool production. Figures 1 and 2 bring into light typical microstructures of the conventionally produced Fig.1. and P/M made ledeburitic steel, Fig.2.
HEAT TREATMENT AND COMPLEMENTARY PROCESSES

The cold work tools made from P/M ledeburitic steels have to be heat treated before use. Heat treatment normally involves vacuum austenitizing, inert gas quenching and several tempering cycles [13,14]. Other media (salt bath, atmosphere) are no longer preferred for the austenitizing or for the quenching.

The austenitizing temperature has to be chosen with respect to the optimal complex of hardness and fracture toughness. The tempering should in any case be realized to the secondary hardness. To process the tools made from the ledeburitic steels, the primary hardness can not be recommended, because a low temperature used for the tempering increases the risk of insufficient reduction of hardening stresses and subsequent cracking [15]. Moreover, this treatment does not allow us to perform the surfacing because of too low a tempering temperature.

The heat treatment increases the hardness usually to 700 - 800 HV, which permits the use of tools in the production line directly. In some cases however, the use of P/M material itself can not lead to the acceptable properties of tools, and post - heat treatment must be used to meet the demands of the end-users of tools [16].

Plasma nitriding was established as a method convenient for a final treatment of the material [17,18] or as a pre-treatment prior the PVD – coating [19,20]. It is due to the fact that the main parameters of plasma nitriding (temperature, dwell time, atmosphere constitution) can be very accurately adjusted and they are closely related to the properties of nitrided layers. To meet the requirements on a plasma nitrided region, the following phenomena must be achieved:
1) Sufficient surface hardness increase in comparison with the core material.
2) Formation of nitrides that increase the wear resistance and lower the friction coefficient.
3) Formation of compressive stresses in diffusion layer.

![Figs.3 and 4. Microstructure of nitrided region on P/M ledeburitic steel, overview (left), detail (right).](image)

The application of the plasma nitriding can easily increase the hardness of the ledeburitic steels, although they differ one from an other in the chemical composition and thereby in nitriding capability [21]. Figures 3 and 4 show the nitrided layer developed on the cold work steel VANADIS 6 with typical features in the near-surface area. Also the formation of nitrides in the near – surface region and the lowering of the friction coefficient was clearly demonstrated in papers published recently [22,23].
The compressive stresses formed due to the nitriding can reach up to very high values, typically of a 1000 MPa [24]. They can successfully compensate the tensile component of the cyclic fatigue loading and prevent the fatigue failure [25]. In these cases, the service — life of tools can be prolonged many times which leads to significant cost savings. Improved fatigue — life time is, unfortunately, connected only with the presence of the nitrided region on the surface, and after this region is removed, for example by wear, its beneficial effect vanishes.

Nevertheless, the nitrided region formed in the near-surface area does not have only positive effects. As published recently [26], a brittle region is formed on the surface. This region makes a serious lowering of the fracture toughness on the steel since it has a limited resistance against cracking, and when it cracks, it does so without any plastic deformation. Figure 5 demonstrates the lowering of the three point bending strength as a function of the nitriding parameters. Figure 6 brings out a typical example of the nitrided region cracked during the three point bending test, with typical cleavage facettes. The lowering of the fracture toughness is more dramatic as the thickness of the nitrided region increases. But not only the thickness itself, but also the higher nitrogen saturation, presence of a larger amount of nitrides etc., lower the fracture toughness. Therefore, the nitriding of thin – walled or fine tools can not bring any beneficial effect and can not be recommended, or may be performed only under strongly determined conditions.

![Fig.5. Three point bending strength of the Vanadis 6 steel as a function of the nitriding parameters.](image5.png)

![Fig.6. Cleavage region on the surface.](image6.png)

To minimize the undesirable lowering of the fracture toughness, the saturation of the surface with nitrogen has to be optimized, well controlled and the thickness of that area must be restricted only to a necessary value. In this case, however, the risk of wear rises and the surface has to be protected with an alternative method.

The hard layers, developed by various PVD – methods [24,27,28], can easily meet the last requirement. They have an extremely high hardness, usually exceeding 2000 HV, lower the friction coefficient considerably and can protect the tool surface against wear. On the other hand, the PVD – layers differ significantly from the steel substrate as in the physical properties (Young modulus, coefficient of the linear elongation…) just as in the mechanical properties (hardness, toughness etc.). In heavily loaded systems in particular, they behave in a completely different way, compared to the substrate, and may easily be damaged.
The efforts leading to the development of so-called duplex layers are thus logical. The presence of the inter-layer, formed by the plasma nitriding, has a supporting effect on the PVD-overlay. On the other hand, the PVD-layer protects the tool, including the nitrided region, against wear and it retains the beneficial effect such as the existence of compressive stresses etc. A typical example of the duplex-layer is demonstrated in Fig. 7. It can be said that the PVD-layer and the nitrided region work simultaneously, in a synergic system, whereas one without the other does not function well. The beneficial effect of this synergy was already pointed out [29,30].

![Fig.7. The duplex-layer (CrN + plasma nitriding) on the Vanadis 6 steel.](Image)

Figure 8 shows a typical failure of a tool (punch) working under a cyclic strain such as a medium cycle fatigue. The tool was damaged after cca 3 100 cycles. As clearly shown, the fracture was initiated at more places at the surface and propagated throughout the material in a low-energy plastic manner, which is typical for high strength steels, Fig.9. The situation in the case of a plasma nitrided punch differs clearly from that for the no-nitrided punch. It is evident that the fracture is initiated at a certain depth below the surface and not at the surface (Fig.10, arrow designated). This depth generally corresponds approximately to 75% of the total thickness of the nitrided layer. The fracture surface of the core material can be described similarly to that of the no-nitrided material, Fig.11.

The nitriding brought on a prolonging of the life-time but only up to the moment when this region is removed from the substrate, mainly due to wear. The period for the removal can vary in a relatively wide range and also depends on many factors that can not be easily determined – in general they can be designated as “working conditions”. Figures 12 and 13 show the life-time of the tools of the same design, but they worked in different places of the follow die. The life-time of the tools working in the rear position was improved by means of the plasma nitriding many times and became stable over 30 000 pieces made. On the other hand, the tools working in the frontal position caused many difficulties – the life-time was not stable and in many cases insufficiently short for the end-user. The reasons for this instability were: the embrittlement of the surface via the plasma nitriding and also wear. Unfortunately, the harder and thicker the nitrided layer, the material becomes more brittle – therefore the complex of demands given by the end-user can not be met only via plasma nitriding.
We tried the duplex – coating, but the plasma nitrided inter – layer was formed at 470°C for 30 min and not 500°C/60 min, in order to reduce the surface embrittlement to a minimum. The results shown in Fig.14 indicate that the life-time was stabilized in the range 7500 – 19000 pieces made, i.e. the follow die worked at least one relay without the requirements of the tools removal, which satisfied the end-user of the technique completely.
Fig.14. The lifetime of punches in the “frontal” position, duplex-coating with TiN + nitriding 470°C/30 min.

SUMMARY

The overview – paper describes a promising processing route that can overcome some of the problems that occur in tooling. What is pointed out is that the P/M of the rapidly solidified particles insures a high homogeneity and isotropy of the material? This creates a good background for production stability when the tool made from the P/M steel is introduced into the production line directly. If any further requirements are made for the tools, then there is the surface technique consisting of the plasma nitriding and the PVD – layering, which can successfully be applied to meet these demands. In this case, the components of so – called duplex-layer work in a synergetic system, where the plasma nitrided layer acts as support for thin PVD – overlay, increases the fatigue life – time and the PVD – overlay protects the tool against wear.

REFERENCES

Engineering Conference in Europe, Gothenburg, Sweden, 2000, p. 197


