Investigation on heat treatment of powder metallurgy carbon free Fe-Co-Mo alloy

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Carbon free Fe-Co-Mo alloys are relatively new cutting materials characterized by improved thermal stability.
In general, tools materials used for cutting applications, have to be abrasive and wear resistant and additionally they exhibit high hardness at elevated temperature in order to withstand the thermal loading of the cutting edge which leads to softening of the tool.
The ternary system Fe-Co-Mo exhibits as-heat treated hardness levels similar to those of high speed steels but with higher resistance to heat softening due to high thermal stability and thermal conductivity. For this reason, this alloy can be used in the fabrication of machining tools for application where high temperatures and stresses due to temperature changes play a major role, which is the case by machining hobs and mills or machining titanium alloys, Ni – based materials or stainless steels.
Carbon free alloy type Fe-Co-Mo exhibits a precipitation hardening mechanism achieved by solution annealing followed by fast quenching and subsequent aging of the Fe-Co-Mo matrix at temperatures below austenite transition to form very hard nm-sized intermetallic precipitates.
In this investigation, iron based Fe-25 wt.- % Co-15wt.-% Mo alloy BÖHLER MC 90 Intermet has been used to test different heat treatment parameters, such as temperature and quenching pressure, in order to study their effects on microstructure and mechanical properties in terms of hardness.

KEYWORDS: BÖHLER MC 90 INTERMET, CARBON FREE FE-CO-MO ALLOY, VACUUM HEAT TREATMENT, PRECIPITATION HARDENING, MICROSTRUCTURE, HARDNESS.

INTRODUCTION

High performance machining operations, especially metal cutting applications, require reliable and durable tools characterized by good combinations between strength and toughness. Different groups of tool steels have been designed for the manufacturing of cutting tools [1]. In the annealed state, tool steels are soft and machinable to the desired shape but they exhibit high strength, high hardness and high wear resistance as a consequence of the heat treatment of the tooling parts.
Nowadays, high-speed tool steels are used for most of the common types of cutting tools including drills, reamers, taps, milling cutters, end mills, hobs, saws, and broaches.
Their higher mechanical properties, such as abrasive resistance and hot hardness, are due to the presence of carbide forming elements as tungsten, vanadium, cobalt and molybdenum. The more severe the service condition (higher temperature, abrasiveness, corrosiveness and mechanical loading), the higher the alloy content and consequent amount of carbides required for the tool steel.
The hardening mechanism of high speed steels is connected to the precipitation of secondary – hardening carbides during the heat treatment resulting in a high performance cutting tool for service temperatures up to about 600°C. Anyway, at higher temperature, a loss of cutting performance can be observed as a consequence of the rapid over aging of carbides.
For this reason, for higher operating temperatures low cutting speeds and cooling lubricants must be used.
This undesired phenomena is particularly observed in case of difficult machining titanium alloys and nickel alloys. Those tough materials are very difficult to machine, since the thermal softening combined with high abrasive wear at the cut-

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ting edge can lead to the premature failure of the cutting tool during application.
A promising alternative to increase cutting parameters and productivity is represented by the use of the carbon free cutting material BÖHLER MC 90 Intermet produced through a powder metallurgical process. This iron based alloy Fe- 25 wt.-%Co-15wt.-%Mo is characterized by superior thermal properties, such as thermal stability and thermal conductivity, when compared to traditional high speed steels, resulting in an improved service life for the tool. For these features, the BÖHLER MC 90 Intermet is particularly suitable in case of difficult to cut materials, such as stainless steel, nickel alloys and titanium alloys.
Nowadays, the carbon free alloy Fe-Co-Mo is mainly used in the automotive industry for dry machining of internal gears. It has been demonstrated that the use of the Fe-Co-Mo alloy instead of high speed steels made it possible to increase the cutting parameters and therefore increase the productivity [2]. For this reasons, Fe-Co-Mo alloy is used in the manufacturing process of several hobs and end mills geometries.
In the ternary system Fe-Co-Mo, the hardening mechanism is due to precipitation of very fine intermetallic phases from the matrix, the so called μ-phase $(Fe, Co)_7Mo_6$.Thanks to these fine precipitates, hardness values up to 68 HRc and high thermal stability are obtained. The hardening behaviour of Fe-Co-Mo alloys is similar to Al – alloys. After solution annealing and quenching the Fe-Co-Mo martensitic matrix is quite soft (~ 45 HRc) but its hardness increases with ageing process at elevated temperatures below austenite transition due to the formation of the intermetallic μ-phase precipitates [3,4].
The low hardness after solution annealing allows machining of tools after quenching and the final aging step takes place without dimensional changes or distortion of components as it doesn't involve any phase transformation. Therefore it can be said that geometrical precision after hardening is higher for the ternary system Fe-Co-Mo when compared to the case of common high speed steels.
The aim of this study is to investigate the effects of different heat treatment parameters on microstructure and hardness of the PM-alloy BÖHLER MC 90 Intermet.

EXPERIMENTAL

All experimental heat treatments have been carried out on a Fe-Co-Mo alloy with the nominal composition shown in table 1.

	Fe	Co	Mo	Si	C
Wt.-%	Bal.	24.9	14.9	0.01	0.03

Tab. 1 - Nominal composition of Böhler MC 90 Intermet in supplied condition

From the bar of 82 mm diameter furnished by Böhler Edelsthal, voestalpine division of High Performance Metals Italia, different disc – shaped heat treatments samples of about 22 mm thickness have been cut.
For control purpose, hardness test has been performed in the as – delivered state before the execution of the heat treatment. The hardness test has been carried out using a Vickers testing machine with an applied load of 20 kg. The measured hardness in the as-delivered state was 380 HV20.
A light microscope image of the microstructure in the as – delivered state is shown in Fig. 1

Fig. 1 - MC 90 Intermet microstructure in longitudinal direction prior to the heat treatment (500x)

As confirmed by the performed EDS analysis, the material in the as – delivery conditions contains a certain amount of coarse μ phase due to the manufacturing process of the material itself. In any case, the microstructural homogeneity is guaranteed.
The BÖHLER MC 90 Intermet specimens have been heat treated in horizontal vacuum furnaces equipped with high pressure Nitrogen gas quenching system and convection technology. Every heat treatment cycle has been carried out in presence of a load sensor inserted in an appropriate heat sink in order to monitor the temperature during the entire process.
To study the effect of temperature selection during the solution annealing, the austenitization has been carried out at different temperatures in vacuum condition, varying from 1170°C to 1195°C for 30 minutes, followed by fast quenching with Nitrogen gas at 5.5 bar pressure, while the aging step has been carried out at the fixed temperature of 590°C for 3 hours in protective atmosphere condition with Nitrogen gas and convection mode.

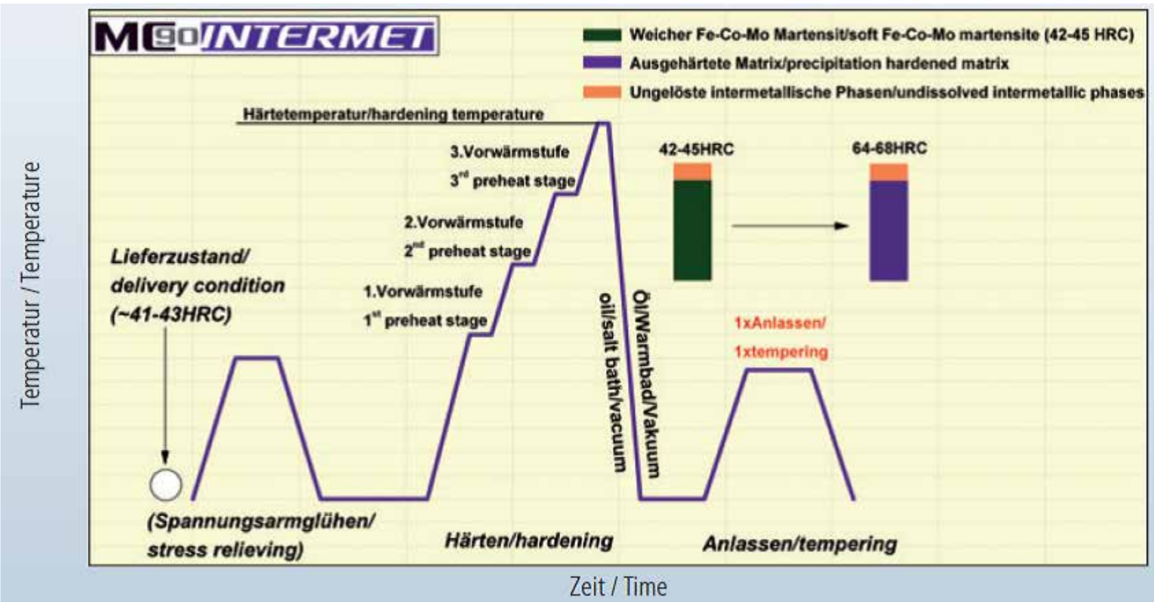

Fig. 2 - Heat treatment process of BÖHLER MC 90 Intermet [5]

For some test pieces, the heat treatment has been carried out at fixed optimal temperatures, i.e solution heat treatment at 1190°C for 30 minutes and aging at 590°C for 3 hours, but varying the Nitrogen gas pressure from 4 to 7 bar during the quenching phase.
In order to study the influence of the stability of the quenched Fe-Co-martensite, aging treatments have been carried out immediately after solution heat treatment and a month later.
Any differences have been found from hardness and microstructure point of view between the samples aged in continuous and aged after a month.
Hardness tests have been performed on each specimen on the surface and in the longitudinal section but, as expected, no differences have been noted. Metallographic preparation and etching with Picric acid have been performed in all cases.

HARDNESS RESULTS

In table 2 the results of hardness measurements after solution heat treatment are shown. Hardness tests have been performed employing both a Vickers- Armstrongs hardness tester and a Future-Tech Rokwell hardness testing machine.

Sample No.	Solution annealing		Vickers hardness HV30	Rockwell hardness HRc
	T [°C]	P [bar]		
1	1190	7	493 HV30	46 – 46.5 HRC
2	1190	4	490 HV30	45.5 – 46.5 HRC
3	1190	5.5	496 HV30	46.1 - 47.1 HRC
4	1195	5.5	496 HV30	45.8 – 46.2 HRC
5	1170	5.5	598 HV30	53.4 – 54.8 HRC

Tab. 2 - Measured hardness values of the different solution heat treated samples

Typical hardness values achievable after solubilisation are in the range within the range of 44 – 47 HRC. All hardness values are compliant with those expected saving the case of solution annealing performed at 1170°C.

In table 3 the results of hardness measurement after solution annealing in different condition and aging at 590°C are reported.

Tab. 3 - Measured hardness values of completed heat treated samples in different conditions

Sample No.	Vickers hardness HV30	Rockwell hardness HRc
1a	950 HV30	67.5 – 68 HRC
2a	905 HV30	65.2 – 66.2 HRC
3a	950 HV30	67.0 HRC
4a	927 HV30	66.0 - 66.8 HRC
5a	644 HV30	57.0 – 57.5 HRC

In the case of series n.1 of specimens, the Vickers hardness impressions have revealed the presence of a brittle microstructure, which is due to the high hardness of 68 HRc, as shown in Fig. 3. This fact, can be related to the very high pressure used during quenching after solution heat treatment.

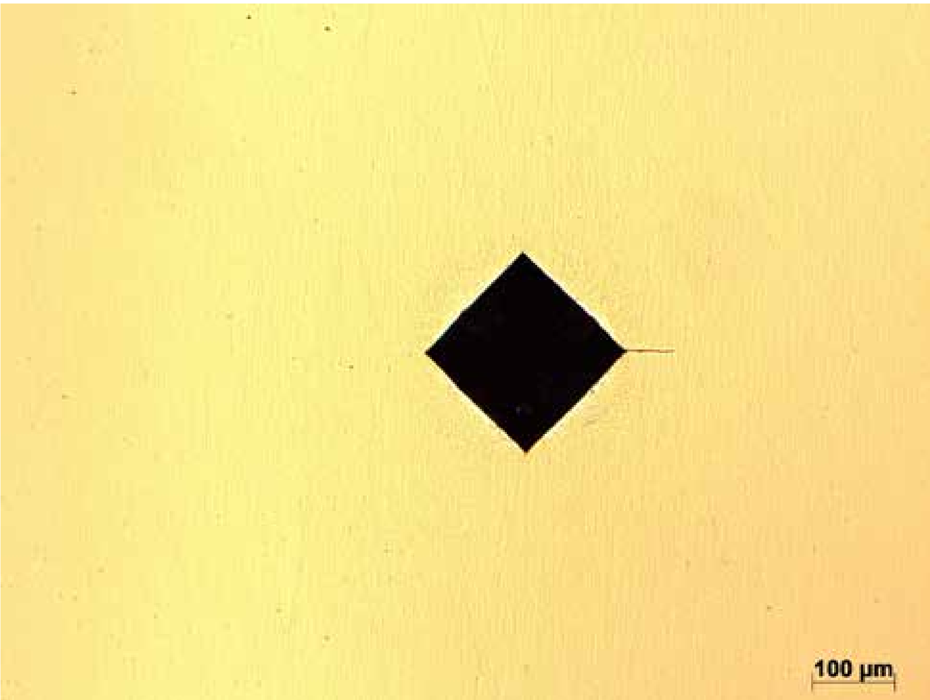

Fig. 3 - Vickers hardness indent in specimen n. 1a, 950 HV30 (100x), micro crack visible on the right end

Hardness testes performed on specimens aged at 590°C a month later of solubilisation with different parameters, have not revealed any substantial differences in hardness values between samples aged in continuous.

MICROSTRUCTURE INVESTIGATION

In table 2 the results of hardness measurements after solution heat treatment are shown. Hardness tests have been performed employing both a Vickers- Armstrongs hardness tester and a Future-Tech Rokwell hardness testing machine.

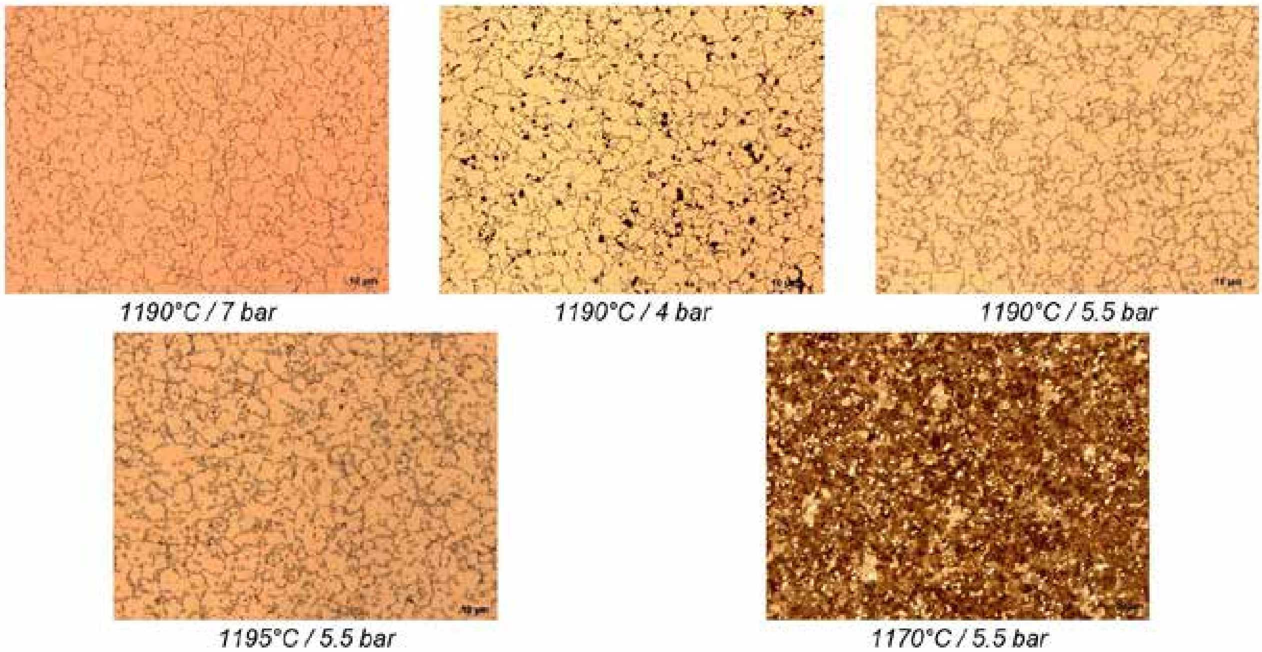

Fig. 4 - MC90 Intermet microstructures after solution annealing with different parameters (500x).

Light microscope images of the microstructures of the specimens after austenitization in various conditions and aging at 590°C are shown in Fig. 5.

Fig. 5 - MC90 Intermet microstructures after solution annealing with different parameters and aging at 590°C.

After solution heat treatment and aging, all samples are characterized by fine and regular microstructure but differences in the amount of precipitates can be observed. All samples contain primary precipitates of μ phase of 1 – 4 μm diameters and secondary precipitates of μ phase, which are mostly responsible for the final hardness, in the nm range, however these cannot be resolved by light optical microscopy.
Differences from microstructural point of view, and, as a consequence, from the hardness one, are related to the austenitization temperature. At higher temperatures, the μm-sized μ-phase particles are nearly completely dissolved in the matrix and, as observed for high speed steel, the grains tend to coarse with subsequent loss of mechanical properties, e.g. ductility.
At lower solution annealing temperature of 1170°C, less μ- particles are dissolved and therefore less molybdenum is available for the subsequent precipitation of intermetallic nm-sized μ-phase particles to guarantee an adequate increase in hardness. The microstructure after solution annealing at 1170°C and gas quenching with 5.5 bar nitrogen, shows the presence of μ-phase precipitates which probably are the same observed in the as – delivery condition. After complete heat treatment, i.e. at the end of aging step at 590°C, no substantial differences in the microstructure have been observed expect for a microstructural refinement.
By that it could be shown that the solution annealing must be carried out in that way to guarantee a small remaining amount of intermetallic μm-sized μ-phase particles in the matrix in order to avoid the coarsening of grains. However, too low solution annealing temperatures lead to lower hardness after aging.

SCANNING ELECTRON MICROSCOPY

Scanning electron microscope combined with Energy Dispersive Spectrometry (SEM – EDS) has been used to evaluate the chemical composition both of the matrix and precipitates from a quality point of view.
An example of SEM image acquired using BSD detector is shown in Fig. 6.

The SEM-image in back-scatter image mode reveals Fe-Co matric and the μm-sized μ-phase particles.
Using the scanning electron microscopy, also the measurement of precipitates' size has been done.

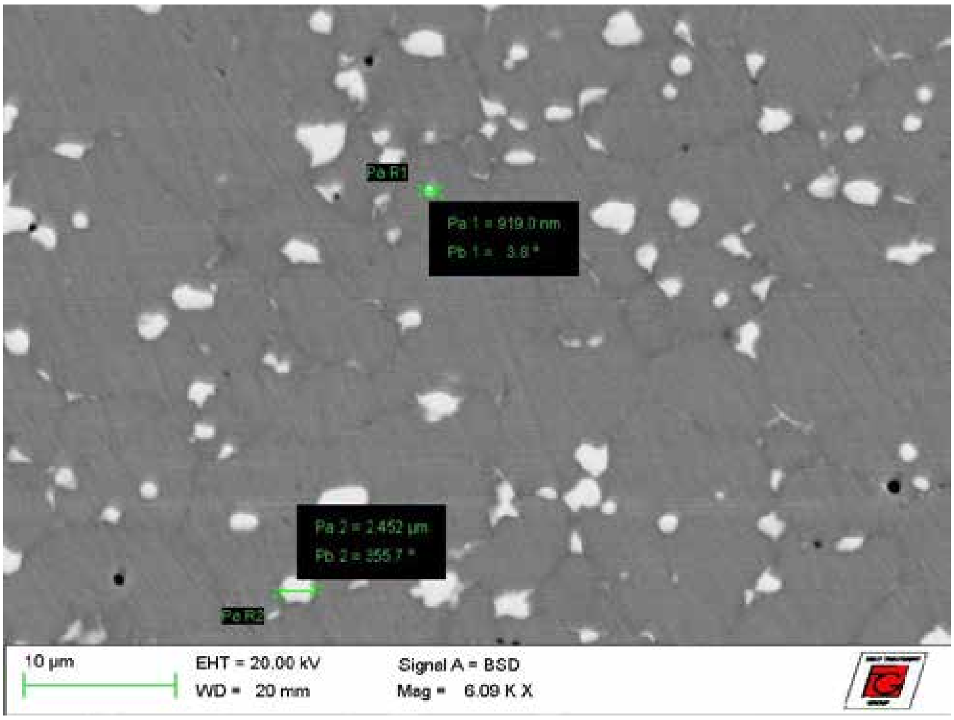

Fig. 6 - Sample n. 3A, solubilized at 1190°C and aged at 590°C

No substantial differences in precipitates dimension between the various heat treated specimens have been found. All aged samples are characterized by primary intermetallic phase with size in the μm range and secondary intermetallic phase with diameters in the nm range. The intermetallic μ-phase particles are mainly located at grain boundaries.

Using the SEM – EDS a martensitic matrix, which appears grey, consisting of Fe – Co with some Mo, has been observed in all fully heat treated specimens while both the primary and secondary precipitates consist of μ - phase with chemical composition $(Fe, Co)_7Mo_6$, as confirmed in previous investigations [6].

CONCLUSION

The studies of the role of solution annealing temperature as well as quenching pressure selection have been performed.
At lower temperature, i.e. 1170°C, the measured hardness is out of the expected value being the amount of dissolved molybdenum after solution annealing not sufficient to guarantee the precipitation of nm-sized μ-phase and, as a consequence, to achieve high hardness values.
At higher temperature, the amount of dissolved molybdenum increases and also the amount of secondary precipitates formed during the aging step. However, the solution temperature of 1195°C is not high enough to appreciate substantial differences in the final hardness values when compared to those obtained at 1190°C, i.e. at optimal solution temperature.
As expected, varying the quenching pressure, differences on final hardness values have been found. Using 4 bar nitrogen gas pressure after austenitization, a maximum of about 66 HRc have been obtained on sample n. 2a while hardness level of 68 HRc has been measured on sample n.1a quenched whit 7 bar nitrogen gas pressure. However, a minimum of 5 bar nitrogen gas pressure must be used to obtain suitable hardness level.
The solution treatment affects the final content of intermetallic μ-phase particles in the final microstructure as well as the grain growth and it should be done at a temperature at which a sufficient amount of molybdenum is dissolved in order to allow a proper subsequent secondary hardening. Moreover, also the quenching step must be carefully performed, selecting the proper gas pressure in order to avoid undesirable effects, , e.g. brittleness effects in case of too high gas pressure or lower hardness at lower gas quenching pressure.

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