Journal of Korean Powder Metallurgy Institute
Vol. 5, No. 4, 1998

Improvments in Cost Reduction for Vacuum Sintering and Vacuum and Overpressure Sintering for Tungsten Carbides

Dieter Ermel
ALD Vacuum Technologies GmbH, D-63526 Erlensee, Germany

(Received October 28, 1998)

Abstract In all larger hardmetal workshops furnaces for dewaxing, vacuum sintering or vacuum and overpressure sintering are today's standard. The furnace technology is well established. Equipment specifications such as operating overpressure, determine sintering cost, product quality, safety and reliability of the furnace and ultimately influence the competitiveness of the hard metal procucer in the global market. Essential furnace requirements are an efficient utilization of the furnace, an environmental friendly dewaxing system, high temperature uniformity, metallurgical treatment with process gases, as well as reduced cooling time by means of rapid cooling. Examples of reduced sintering costs are described achieved using a new design of vacuum sintering furnace with an improved rapid cooling device, cooling times are reduced by up to 45%. Additionally, a cost comparison of two different designs of vacuum overpressure sintering furnaces are included.

1. Cost Reduction for Vaccum Dewaxing and Overpressure Sintering of Tungsten Carbide

Up to three process steps, dewaxing, vacuum sintering and overpressure sintering in the same cycle will be performed in one and the same overpressure sintering furnace.[1)]

Important improvements in mechanical properties of cemented carbides can be seen by using the overpressure sintering process. The next table shows some results of corrosion resistant hardmetal WC+

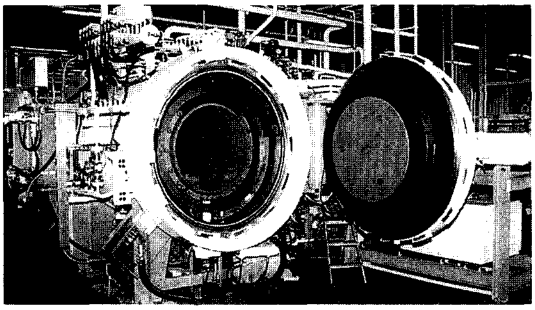


Fig. 1. ALD Furnaces Type VKPgr 50/50/170, 6 MPa, 1600 °C with paraffin dewaxing, after installation at site, capable for a workload of more than 1000 kg tungsten carbide parts.

Sintering Process		Density g/cm³	Magn. Sat. 4Πσ	Hardness HV	Binderlaking >4 μm/cm²	Porosilty
40 min 1450℃ Vacuum		13.97	0.6	1375-1386	400	>A08>B02 CO4
40 min 1450℃ Sinter/HIP	2 MPa Ar	14.29	0.03	1487-1526	Ø	A00>B02>C06 3 pores to 75 μm/cm²
40 min 1450℃ Sinter/HIP	4 MPa Ar	14.30	0.22	1500-1509	Ø	A00>B02>C04
40 min 1450℃ Sinter/HIP	6 MPa Ar	14.41	0.42	1522-1548	Ø	A00>B00<C04
40 min 1450℃ Vacuum+HIP	>100 MPa Ar	14.14	4.0	1634-1639	1500	A00>B02<C04

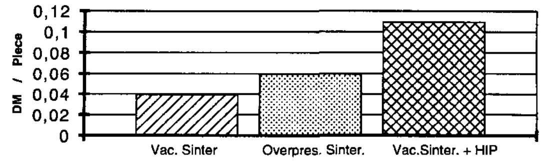


Fig. 2. Production cost for WC-6% Co parts, weight 7.5 g, FPM-Symposium Hagen 12/87.

Cr+Ni.

Also remarkable production cost savings are achieved when compared to dewaxing and sintering in one furnace, followed by post HIP in a separate HIP unit with 100 MPa. A comparison in production cost[2)] is shown in Fig. 2. The value of the sintering pressure during overpressure sintering is an important cost factor. The gas cost for any overpressure sintering furnace carried out with 5 MPa in comparison to 10 MPa will be 50% less. Further, the opinion as higher the pressure as better the quality of the hardmetal procuct is incorrect. Higher pressure can reduce the quality of the hardmetal product by means of creating so called, cobalt lakes, see Fig. 3, 4 and Table above.

Lueth,[3)] Kieback, Kaysser,[4)] Kim,[5)] have described the phenomenon and given explanation how to determine the critical sintering pressure. The critical sintering pressure during overpressure sintering, which should not be exceeded, depends upon the grain size of the cemented carbide and the mean value of the distance between the carbide particles (Fig. 4). The sintering pressure, applied to the heated workpieces has to be less than the capillary forces which predominate in the liquid phase of the cobalt metal binder. Once the argon gas pressure exceeds the capillary forces, the cobalt will be forced into the pores and can not be eliminated. These so called cobalt lakes will remain in the hardmetal and are undersirable for the marterial properties of the hardmetal product.

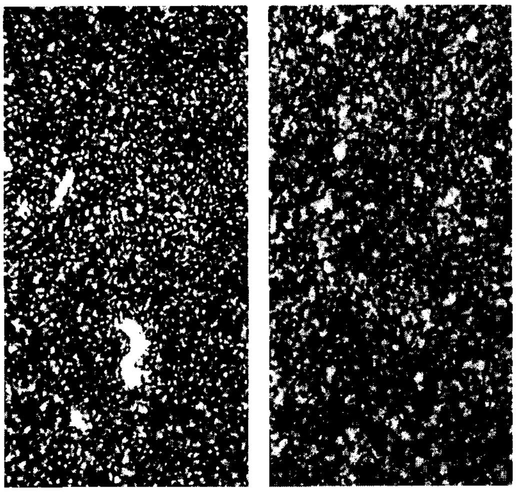
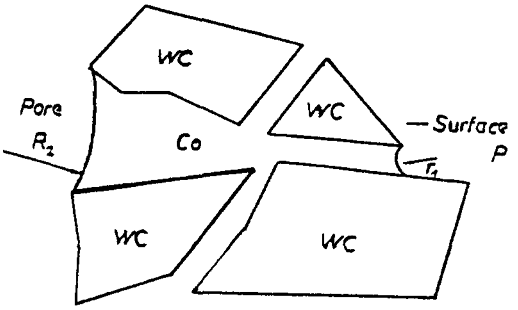


Fig. 3. Grade WC+6% Co, Fig. 3, left, with Co-lakes (vacuum sintered+Post HIP 100 MPa), right, (vacuum and overpressure sintered with >6 MPa) without Co-lakes.



$$P_{krit} = 2\left[1/r_1 - 1/R_2\right]$$

Fig. 4. Influence of the capillary forces during sintering and calculation of the critical pressure (US Patent: 4,591,481).

The following table illustrates some typical value for critical pressures.

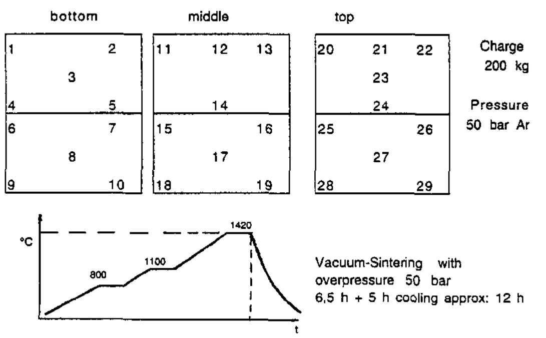
On the other hand the sintering gas pressure must be high enough to remove the residual remaining porosity in this PM material. Lueth,[3)] Kolaska, Dreyer, Schaaf,[6)] have made extensive tests with different hardmetal grades and found that 5.5 MPa is sufficient to get good material qualities, even also for ultra fine grain hardmetal grades. Other important points leading to cost reductions are improvement in the hard and software of the furnace itself which reduce investment cost. A multi-dewaxing system for 3 different dewaxing processes in one system gas been developed and successfully introduced in

Weight % Co	rl	Grainsize	P crit.
6%	0,083 μ	2,5 micron	24 MPa
6%	0,333 μ	10 〃	6 MPa
10%	0,133 μ	2,5 micron	14,4 MPa
10%	0,555 μ	10 〃	3,6 MPa
16%	0,222 μ	2,5 micron	9,0 MPa
16%	0,95 μ	10 〃	2,2 MPa
25%	0,30 μ	2,5 micron	6,5 MPa
25%	1,25 μ	11 〃	1,6 MPa

the hardmetal industry. Paraffin dewaxing with low pressure, paraffin dewaxing with streaming hydrogen at a slight overpressure (1050 mbar abs.), and PEG dewaxing, also under streaming hydrogen with 1050 mbar abs. can be processed in a single dewaxing unit. In the past up to 3 different dewaxing systems were necessary.[7)]

The three independent controlled heating circuits together with a cylindrical inner graphite muffle and a special cylindrical insulation (see Fig. 1) guarantees a temperature uniformity of better than +/- 7 K in vacuum and less than +/-10 K in 5,5 MPa argon.

Sample	HC [Oe]	Sample	HC [Oe]
1	275	16	275
2	275	17	280
3	276	18	273
4	274	19	275
5	275	20	274
6	275	21	276
7	275	22	276
8	275	23	278
9	273	24	278
10	273	25	278
11	276	26	276
12	275	27	278
13	275	28	276
14	276	29	278
15	275		



Vacuum-Sintering with overpressure 50 bar
6,5 h + 5 h cooling approx: 12 h

Fig. 5. Coercitive force as a measure of the temperture distribution in a VKPgr 50/50/90 furnace.

Fig. 5 shows the distribution of 20 samples (K 20 according to ISO 513) and the measured values in Hc (Oe). The measured delta in Hc is 7 Oerstedt. This corresponds to +/- 1.3%, which is much better, than published values in all side heated 3-chamber furnaces with +/- 3%.

This high temperture uniformity over many years service increases the product quality, and reduces any scrap cost for the furnace user. The standardisation to 4 industrial sizes for 81, 225, 375, 425 litres net volume with different options and accessories[9)] reduces the production cost for the furnaces. A compact furnace design, the summary of sub-assembly units, the high pre-assembly standard of these furnaces reduces the production cost as well, and the cost for assembly, start up and commissioning on site an reduces the investment and installation cost.

Today more than 63 such VKPgr overpressure furnaces have been sold worldwide. Quality assurance according to ISO 9001 guarantees the hightest standard in quality of the manufacturer of the equipment and service. The VKP furnace has 2 different integrated cooling systems, see Fig. 6 : the normal fast cooling device within the pressure vessel, the large "cooling jacket" and an additional rapid cooling system with argon refilling during cooling. Both systems incur no extra investment cost. No external high presure heat exchanger or internal motor driven blower are necessary.[10)] For the rapid cooling, only a certain amount of additional argon gas (up to 50% of the normal gas volume) is required. Generally speaking, the cost for the highen gas consumption is easily offset by reduction in cycle time, higher

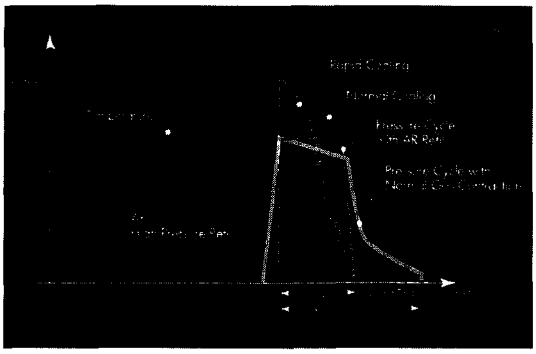


Fig. 6. Schematic vacuum and overpressure sintering cycle with normal fast cooling (4,1 h) and with rapid cooling with argon gas refilling during cooling (2,5 h), furnace type VKPgr 30/30/90.

productivity with the same furnace and better flexibility in production time.

A comparison for 2 nominally similar vacuum and overpressure furnaces follows. Price, pressure and temperature were equal, but remarkable differences in productivity and earnings have been found after more detailed consideration of factors like useful net volume, heat-up time and fast cool down time. Calculation is based on cycles without dewaxing.

It can be seen that furnace B has a much higher output than furnace A. Furnace B produces 2,38 Mio DM/year more hardmetal than furnace A. Any decision to select the right furnace for this type of production should be not only be based on nominal measures and price. More important is the higher productivity, which helps for faster return of investment.

2. Cost Reduction for Vacuum Dewaxing and Vacuum Sintering of Hardmetals

A long recession in the hardmetal industry has delayed developments in this area. However, improved business conditions have led to new ideas and the

Furnace		A	B	Δ
nominal net volume	[l]	81	81	0
physical useful volume	[l]	140	183	43
installed electrical power	[kVA]	150	350	200
heat-up time to 1420℃	[h]	5,9	3,9	2
fast cool down time	[h]	5,6	4,1	1,5
cycle time (without dewaxing)	[h]	20	17	3
cycle/year, 3 shift		210	247	37
capacity in hm/cycle (1,5 kg hm/Vol.1)	[kg]	210	275	65
capacity in hm/cycle (3 shift operation)	[t]	44,1	67,92	23,8
capacity hm/year (3 shift, 100 DM/kg hm)	[Mio DM]	4,41	**6,79**	**2,38**

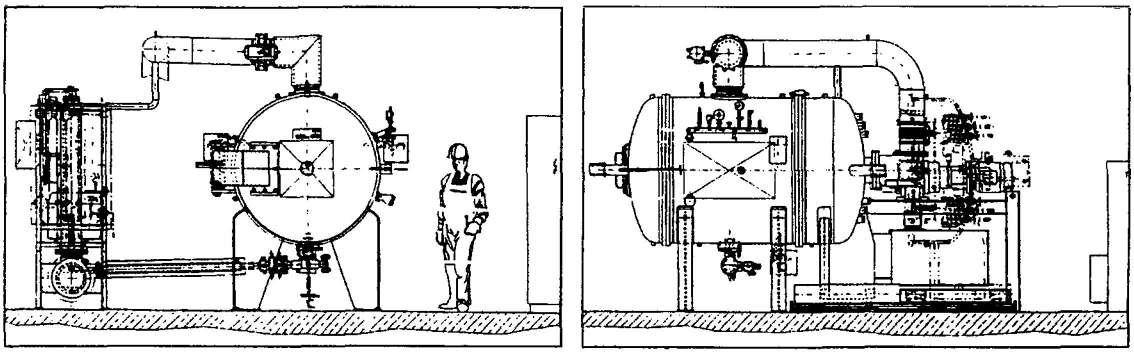


Fig. 7. Vacuum Dewaxing and Sintering Furnace Type VKUgr 60/60/100, with rapid cooling, rearside of the furnace is also shown in open position.

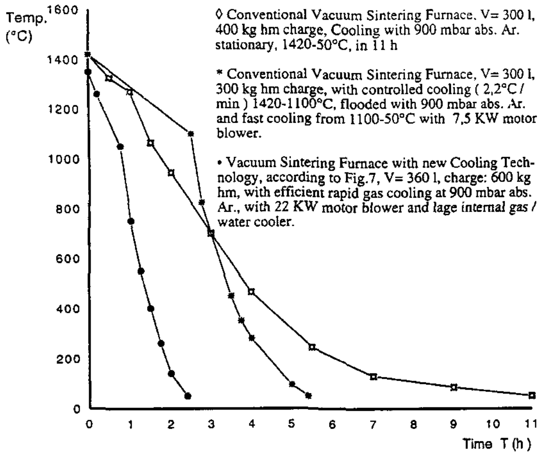


Fig. 8. Comparison of different cooling systems in Cemented Carbide Vacuum Sintering Furnaces, TypeVKUgr.

introduction of an improved furnace design, type VKUgr for net volumes of 110, 360, and 540 litres. This horizontal single chamber furnaces has a better temperature uniformity of up to +/- 3 K, shorter cycle time, resulting from faster heat up and rapid cooling, and the possibility to introduce process gases like N_2, H_2, CH_4, CO, or Ar for metlalurgical reasons.

The proven multi dewaxer for up to three different dewaxing processes from the overpressure sintering furnace VKPgr will be also used with this new furnace. However, the distinguishing design feature of this furnace, over its contemparies, is the new advanced rapid cooling system. Fig. 7 shows this new furnace.

A large gas/water heat exchanger and a blower are mounted inside the rear of the furnace, which can remove the heat of the sorkload rapidly or slowly from the workload if required. The blower is driven externally by means of a 22 KW electrical motor. Since it is only necessary to flood with inert gas up to 900 mbar abs., no special pressure vessel code requirements are necessary.

3. Outlook

It is a long held wish of the hardmetal producers to control the carbon content in the hardmetal during sintering in a reproducible manner. In the vacuum heat treatment of steels,[11,12)] this control of the carbon content is in daily use, in the vacuum carburizing and high pressure gas quenching of automobile gears for example.

A simple and reproducible carbon control during vacuum sintering for cemented carbides could reduce the cost for hardmetal produced in overpressure sintering or vacuum sintering furnaces dramatically.

Developments which would adapt this technology to the above mentioned furnaces are already under way.

References

1. D. Ermel: New Developments in Vacuum and Overpressure Sintering at ALD Vacuum Technologies GmbH, *Proceedings of PM EURO 96*, Stockholm, 1996. p. 127-134.
2. S. Boneff: *Symposium "Sinter/HIP-Technologie"* Hagen, Germany, 1987.
3. R. C. Lueth: Modless Hot Pressing of Cemented Carides, *ADVANCES IN HARD METAL PRODUCTION*, MPR-Conf. 7-9. Nov. Luzern, 1983, p. 13/3-13/11. United States Patent Nbr.: 4,591, 481; May27, 1986.
4. B.F. Kieback, W.A. Kaysser u.a.: Mechanismen der porenausheilung beim Fl ssigphasen-Drucksintern von Hartmetallen, 9. Int. PM Tagung 23-25.10.89, Dresden, Germany, ZFW der ADW der DDR, Dresden Max-Planck-Inst. f. Metallforschung, Stuttgart, Tagungsband 2, S. 85-100.
5. Kwan-Heyeong Kim, Suk-Joong L. Kang, *Proceeding of 2nd. Pacific Rim Int. Conf. on Advanced Materials and Procesing*, 1995, p. 389-394.
6. H. Kolaska, K. Dreyer, G. Schaaf: Use of Combined Sintering HIP Process in the Production of Hardmetals and Ceramics, PMI vol. **21**, no. 1, 1989, p. 22-28.
7. D. Ermel, Fortschritte zur Kostensenkung beim Hartmetallsintern mit Vakuumsiter-und Vakuum-Überdrucksinterofen, 14th Plansee Seminar, HM 45, Volume 2, p. 440-451.
8. R. Bauer: Verfahren und Vakuumanlagen zum Uberdrucksintern von Metall und Keramick, Leybold Durferrit Technische Mitteilungen, Nr.: 155/1.93/2000.
9. Pamphlet of the ALD Vacuum Technologies GmbH, Erlensee, Germany, Vacuum-Equipment VKPgr Dewaxing, Sintering and Overpressure Sintering, Nbr: W0008e/6.98/2000.
10. MPR, ALD furnaces offer rapid cooling to cut cycle

times, MPR Sept. 1996, p. 28-29.

11. Technical Information of the ALD Vacuum Technologies, F. Preisser K. Loser, G. Schmitt, R. Seemann, Vacuum-Based Carburizing Processes with High-Pressure Gas Quenching, ALD-Information, W2005e/9.97/2000/St.
12. Technical Information of the ALD Vacuum Technologies, F. Schnatbaum, A. Melber, Plasmacarburizing of steel in pulsed DC glow discharges, W2001e/1,97/2000/T&D