Modern methods for the production of metal powders by gas atomisation

There are a number of melting and gas atomisation techniques used for powder manufacturing of high alloyed steels, superalloys, reactive and refractory metals. The combination of vacuum melting and inert gas atomisation are required for spherical, high quality metal powders with low impurity content. As energy and environmental costs rise, use of inert gas recycling is increasingly used. For ultra clean powders, use of melting without a crucible is now available on an industrial scale.

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There are many different ways to produce metallic powders on an industrial scale. The most significant are simple milling or crushing processes which produce strictly non-spherical powders, water atomisation processes with more spherical particles but which have significant amounts of oxygen in the powder, and inert gas atomisation processes which provide both, spherical powders and very low impurity levels. Typical shapes are shown in *figure 1*.

Other minor methods include centrifugal atomisation and ultrasonic wave atomisation, but milling, water and gas atomisation are the most relevant technologies in terms of annual produced tonnage. In total about 1Mtpa of powder are produced with these three processes. Milling and water atomisation processes are mainly used for the production of non or low alloyed metal powders (e.g. pure iron powder), where a spherical shape is not required, or where there are low requirements regarding impurity content. For high quality materials with strict requirements regarding shape and impurity level, only the inert gas atomisation can provide suitable product quality.

INERT GAS ATOMISATION

Inert gas atomisation is the leading powder making process for the production of high grade metal powders with specific quality criteria such as spherical shape, high flow rate and rapid solidification structures. Further improvement in powder quality can be achieved by combination of vacuum melting and the use of inert gases as the atomisation medium, leading to low oxygen content and high cleanliness. These special features generally cannot be met by other processing methods.

Typical applications for such high quality powders are Metal Injection Moulding(MIM), plasma spraying and compaction processes for high alloyed materials. Depending on the kind of material to be produced, different melting technologies have to be used in combination with inert gas atomisation.

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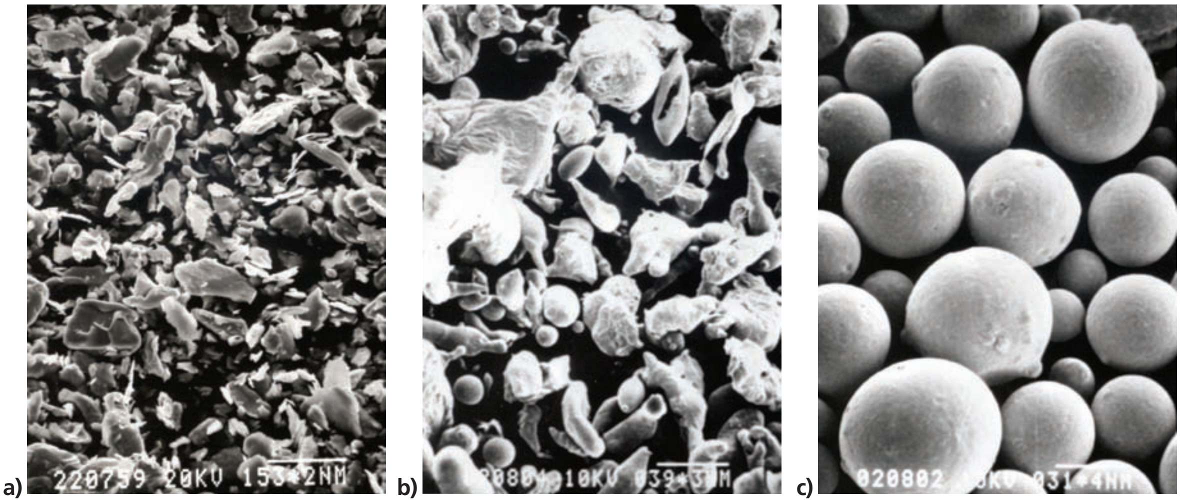

▲ **Fig.1 Typical powder shapes, a) - milling , b) - water atomisation, c) - inert gas atomisation**

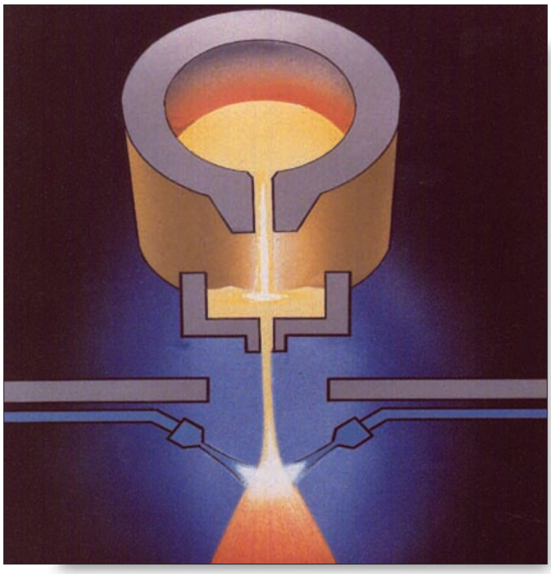

Fig.2 **Principle of gas atomisation (free fall)**

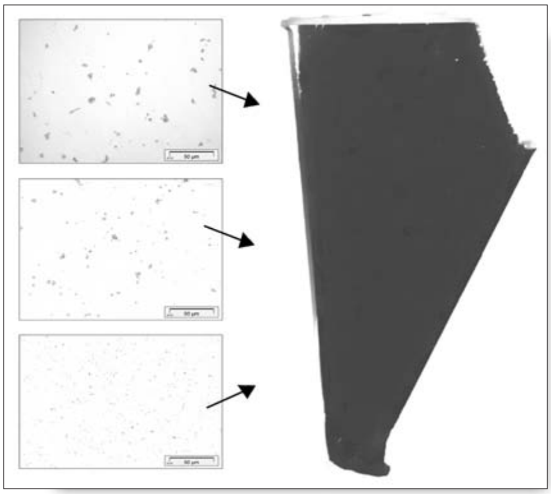

Fig.3 **Clogged atomisation nozzle**

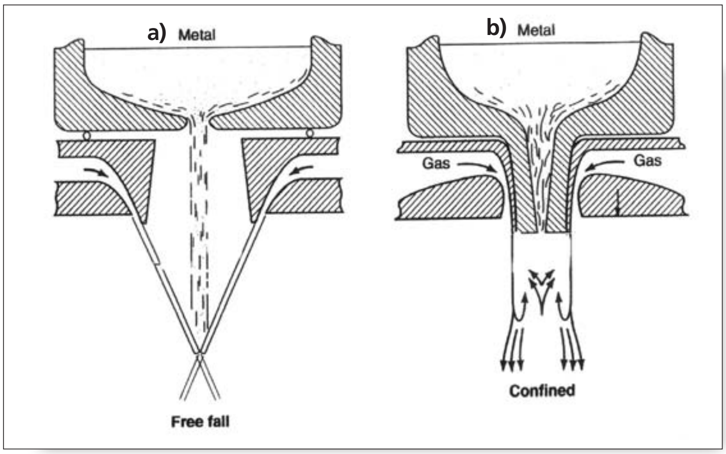

Fig.4 **Different gas atomisation types, a) free fall, b) close coupled**

VACUUM INDUCTION MELTING GAS ATOMISATION (VIGA)

In the VIGA process a metallic material is melted in a ceramic or graphite crucible. Charging, sampling, alloying and temperature measurement are all done under vacuum or controlled inert gas conditions. After the refining and superheating stage the melt is transferred to a tundish from which a defined stream of molten metal is released via a ceramic nozzle. The liquid metal is disintegrated by the kinetic energy of a high pressure inert gas stream.

The formed metal droplets solidify in flight in the atomisation tower located directly underneath the atomisation nozzle *(see Fig.2)*, then the powder/gas mixture is transported via a conveying tube to the cyclone where the powder fractions are separated from the atomisation gas.

The metal powder is collected in a sealed container which is located directly below the cyclone, and is then classified into several fractions and encapsulated. The atomised powder has to undergo some further treatment and classification prior to final compaction in order to achieve a compact material with excellent properties. High grade metal powders used for plasma spray applications require size classification. **[1]**

The orifice diameter of the ceramic nozzle in combination with the metal height inside the tundish determines the metal flow rate. In order to achieve a constant metal flow rate during the whole atomisation process it is quite important that the orifice diameter does not significantly change through erosion or precipitation on the side walls during the run. This is one of the most challenging aspects of the atomisation processes. While erosion of the orifice is related to the type and quality of the ceramic material and the pre-heating procedure of the tundish/orifice system, the reduction of the orifice diameter by inclusions which stick to the side walls depends on several factors. In *figure 3* a clogged nozzle is shown in detail. It is evident that a massive accumulation of inclusions occurred, obstructing proper melt flow. To avoid such an accumulation one has to ensure that these inclusions are not formed during the melting process by using vacuum melting technology in combination with clean feedstock materials.

ATOMISER TYPES

A major factor which influences powder size is the design of the gas nozzle and the type of atomiser. For fine powder (< 45 µm) close coupled systems (sometimes called confined jet systems) are mainly used. For coarser powders (> 100 µm) free fall atomisation is the most common process *(see Fig. 4)*. Here the gas stream directly hits the metal stream and, depending on gas velocity and

Fig.5 **Schematic of a large VIGA plant with a charge weight of 2 x 2t [2]**

nozzle design, different break-up modes for disintegration of the melt stream are established. In the closed coupled atomisation process a wake zone is created by the gas jet directly underneath the ceramic nozzle. This wake leads to fine spreading of the liquid metal film by re-circulation, generating very fine powder at the nozzle tip.

$$d_m = K D \sqrt{\left[\frac{\nu_M}{\nu_G We}\right]\left[1+\frac{m_M}{m_G}\right]}$$

According to Lubanska' s equation the median diameter , d_m, of a powder produced by gas atomisation is related to the Weber number (We), the metal/gas-flow ratio (m_M / m_G), the initial melt stream diameter prior to atomisation (D), the viscosity of metal and gas and to a specific constant (K).

It' s evident from this equation that the Weber number and the initial melt stream diameter have a large influence on the median particle size of the powder. Both parameters are influenced by the design of the ceramic orifice as well as of the type of gas nozzle. All other parameters such as viscosity, metal/gas ratio and the constant K, are either material properties or related to the type of atomiser and cannot be varied that much.

The metal/gas ratio can be varied by increasing the gas flow or decreasing the metal flow. For industrial inert gas atomisers the range of typical metal/gas flow ratios is between 0.2 and 0.8 but, as clearly shown in the equation, this massive change in metal/gas flow ratio leads to a change in the median particle size of only a few percent. This means that above a distinct level, increasing gas flow has only a minor effect on median particle size, and only increases the powder costs. Although this equation was established for free fall atomisation, the fundamental relations are also valid for close coupled atomisers, even though the mechanisms in close coupled atomisation are more complex (related to several different atomisation stages). Confined nozzle atomisation is more versatile in producing different particle size distributions.

INDUSTRIAL SCALE ATOMISATION SYSTEMS

In view of the requirement to produce powder in the most economic way, downtime due to relining, cleaning and maintenance work has to be minimised, and production flexibility maximised.

One method to increase the economic efficiency of a production process is to improve productivity by increasing the production capacity. *Figure 5* shows the layout of a modern large scale powder production system for a batch capacity of up to 2 tonnes. The atomiser has a double crucible door arrangement which allows very fast crucible changing; while one crucible is in production the second crucible can be cleaned or relined in the standby position. This minimises the downtime for furnace change operations, and alloy changes can be accomplished easily.

Furthermore, the atomiser is equipped with a redundant tundish system. Each pouring tundish, including the gas nozzle arrangement, is mounted on a tundish cart which allows rapid tundish change in case of nozzle clogging during the run. The main design criterion for high operational flexibility was integration of a separation valve between melt chamber and atomisation tower which enables the atmosphere in the melt chamber to be adjusted independently of the conditions in the powder tower and the subsequent down stream components. With this special feature only the atmosphere in the melt chamber has to be exchanged for charging and cleaning purposes, whereas the atmosphere in the powder tower and the downstream equipment can be maintained.

Very low leakage rates of below 10^{-2} mbar l /sec for the whole melting and atomisation system as well as low operating pressures in the range of 10^{-4} to 10^{-3} mbar during melting process helps to minimize the danger of nozzle clogging and ensure highest

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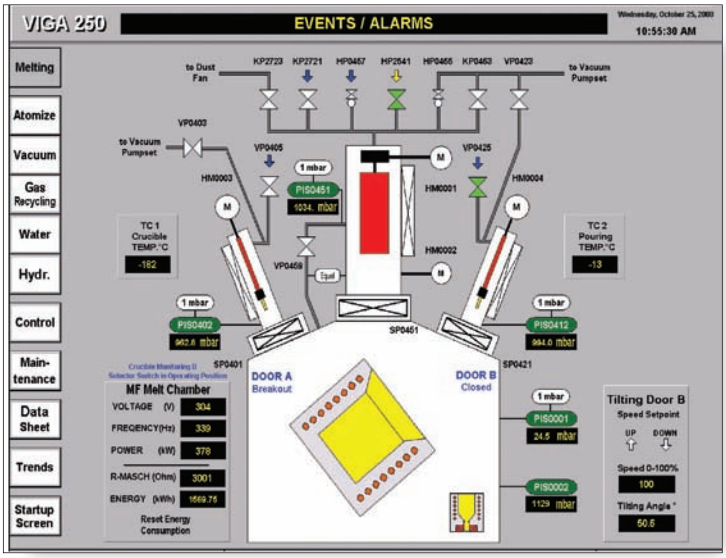


TC 1 Crucible TEMP.°C -182

TC 2 Pouring TEMP.°C -13

PIS0451 1 mbar 1034. mbar

PIS0402 1 mbar 962.6 mbar

PIS0412 1 mbar 994.0 mbar

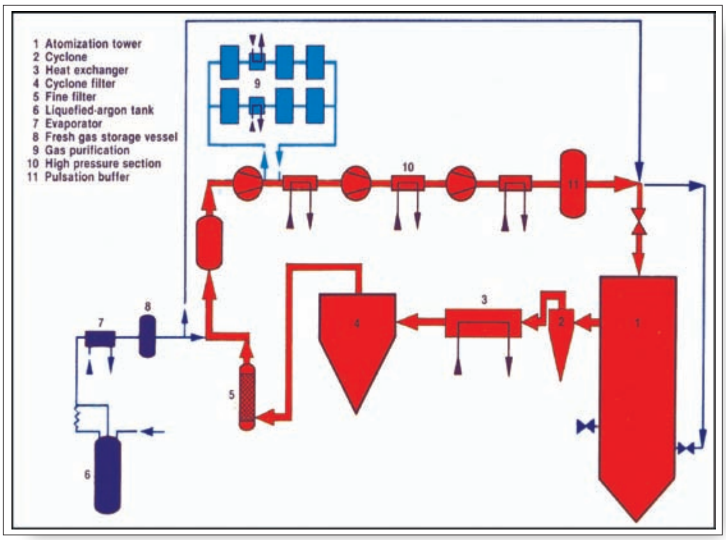
PIS0001 1 mbar 24.5 mbar

PIS0002 1129 mbar

MF Melt Chamber
VOLTAGE (V) 304
FREQENCY(Hz) 339
POWER (kW) 378
R-MASCH (Ohm) 3001
ENERGY (kWh) 1569.75

Tilting Door B
Speed Setpoint
Speed 0-100% 100
Tilting Angle ° 50.6

Ⓐ **Fig.6** **Visualisation of a VIGA system**



Ⓐ **Fig.7** **Inert gas recycling system**

powder quality.

An alternative concept to the conventional Vacuum Induction Melting (VIM) furnace type is a combination of Vacuum Induction Degassing and Pouring (VIDP) and inert gas atomisation. This furnace type can be placed, for example, between two atomisation towers, hence different melts can be atomised alternately between the towers. The molten metal is transferred to the atomisation system via a preheated launder, and a specially designed chamber permits both preheating and changing of the launder as well as the tundish with nozzle arrangement, without the need to vent the system. The strict and thorough separation of the two towers prevents cross-contamination between different products. The result is a metal powder with very low foreign particle content.

Process control To achieve reproducible production, adjustment and control of the following atomisation parameters is most important:

- Melt temperature and superheat
- Metal flow rate
- Nozzle arrangement
- Gas pressure and gas temperature

For all atomisation runs, but especially for those with a duration of up to two hours, it is quite important to keep these parameters constant during the process. To fulfil all control demands the VIGA system is equipped with a specially designed process visualisation and data acquisition system *(see Fig. 6)*. With this the operator can be guided through a complete production cycle. The data acquisition system collects a complete information log of the production process and the process data are stored in a database for further analysis. Long term trends as well as short term process analysis can be carried out.

Inert gas recycling A metal powder unit cost is subject to economies of scale. Most variable cost contributors decrease on large atomisers, but inert gas consumption becomes an ever more significant cost, since nozzle pressure and gas-metal ratio are determined by the atomisation process regardless of batch size.

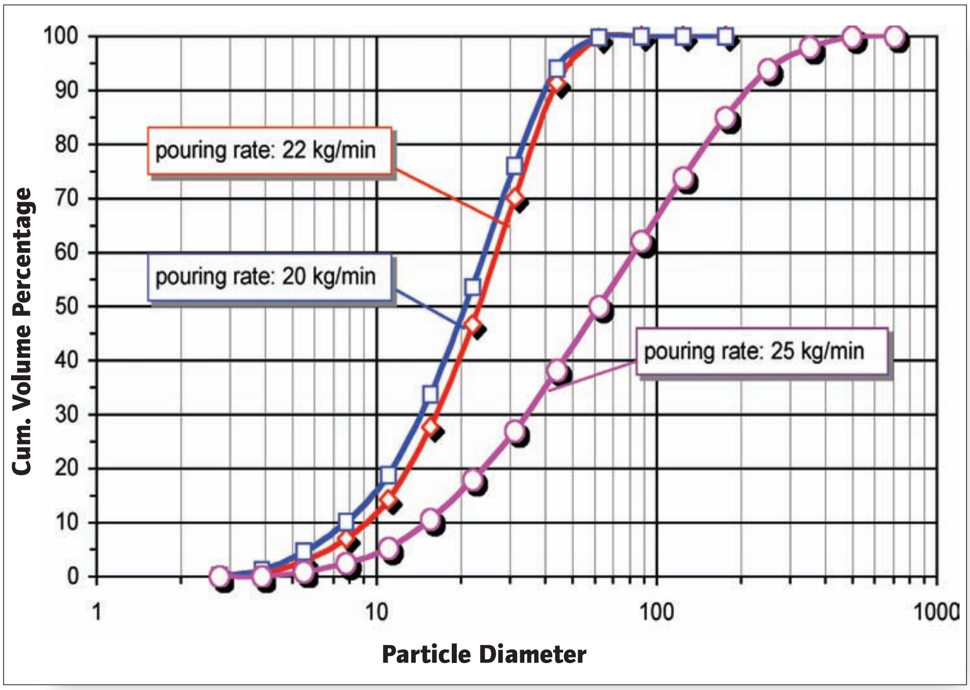
Especially for argon, and to a lesser extent for nitrogen, inert gas recycling is recommended for high production plants. Depending on the required atomization gas pressure, two alternative techniques are available:

- **Recompression** This is applied up to gas pressures of 40 bar and can be used for both nitrogen and argon. The inert gas is collected at atmospheric pressure after the cyclone exhaust and re-circulated to a high pressure gas buffer through dust filters and an inter-cooled two-stage compressor. Due to its greater amount of adiabatic heating, argon requires special compressors with lower compression ratio in each stage when compared to nitrogen. Oil-free, dry-running compressors require less maintenance and hydrocarbon impurity monitoring in the recycling loop.
- **Liquefaction** Liquifaction of argon is applied for gas pressures >40bar because, above that threshold, the compressors required are relatively heavy and expensive. The argon is re-circulated through the cold box, which is effectively a two-stage counter-stream heat exchanger with liquid nitrogen as the cooling agent. In the first stage, the argon is cooled down to

its boiling point by gaseous nitrogen. In the second stage, argon is liquefied by evaporating nitrogen. The liquefied argon is collected in a liquid argon tank from which it passes through cryo-pumps to the high-pressure gas buffer.

In both cases, the recycling efficiency is usually better than 90%. Gas impurities, such as oxygen, carbon dioxide, hydrogen and hydrocarbons can be monitored. A schematic of an inert gas recycling system is shown in *figure 7*.

It is noteworthy that the recycling equipment size and cost is independent of melt/atomisation batch size, being solely determined by the required atomisation gas pressure and flow rate, which are typically in the range of 20 to 60 bar and 1000 to 2000 Sm^3/hour, respectively.



Ⓐ **Fig.8** **Particle size distribution of Ni-based alloys**

PROCESS RESULTS

By using high pressure gas atomisation (up to 60 bar) pre-heating of atomisation gas and due to the fact that the atomisation systems are mostly equipped with a confined nozzle system, a large spectrum of desired particle size distributions can be covered. The degree of cleanliness, the particle shape and particle size distribution are adjustable for various application requirements. The confined atomisation nozzle design allows an easy variation of the powder yield for various kinds of alloys as indicated by *figure 8*.

With regard to the integrated inert gas recycling system the powder can be produced with a minimum oxygen pick up during processing. *Table 1* illustrates the oxygen content of various alloys in the final product after screening the fraction, such as +63/-210 µm.

Grades	**O_2 , ppm**
316L stainless steel	100
High speed steel	55
Ni-based alloy C276	30
Ni-based alloy IN 625	40
Co-based alloy	45

Ⓐ **Table.1** **Oxygen content of various alloys [3]**

CERAMIC-FREE METAL POWDER PRODUCTION

Due to the contact between the melt and the ceramic lining of the crucible the cleanliness level is limited. Even if vacuum melting is applied and the most stable ceramic linings such as zirconia or calcia are used, at very low oxygen contents in the melt the crucible itself acts as a source of oxygen and ceramic particles. Furthermore, the temperature stability of ceramic crucibles is limited to 2100°C and most of the materials which are atomised cannot be melted in a graphite crucible because of carbon pick up. Therefore, highly reactive materials such as titanium, zirconium and hafnium and materials with a melting point beyond 1800°C have to be liquefied without any contact with ceramic or graphite material.

To overcome this problem various solutions were developed, one of the most promising of which is the Electrode Induction Melting Gas Atomisation (EIGA) technique which uses a pre-alloyed rod as feedstock material *(see Fig.9)*. The front end of the electrode is surrounded by an induction coil and, by coupling electric power into the electrode, a melt stream is established which drops into a gas nozzle arrangement where the melt is atomised.

The consumable electrode is continuously fed into the induction coil by means of an electric drive, and the metal flow rate is controlled by the induction power and the feed rate of the electrode. The replacement of the electrode is realised without venting either the melting

Fig.9 Schematic of the EIGA process [4]

chamber or the atomisation tower. The complete process, including removal of the produced powder, is done under inert conditions.

Whereas until now this technique was limited to melt and atomisation rates of 50kg/hour and electrode diameters of 60 mm **[5]**, recently an up-scaling program has been successfully finalised and the process is able to handle electrode diameters up to 100 mm and melt rates above 90 kg/hour in titanium. Based on the experiences made in the up-scaling program a further rise of melt and atomisation rates as well as increasing electrode diameters seem to be feasible.

Up to now titanium, zirconium, other reactive metals and niobium have been atomised by the EIGA process and there seems to be no limit in materials which can be atomised with this technique, including refractory metals with a melting point > 2500°C.

SUMMARY

The combination of vacuum melting and inert gas atomisation offers the most suitable way to produce metal powders which meet the highest quality requirements. Modern atomisation systems have to ensure that fine powder yield is sufficiently high and that contamination by impurities is at very low levels. The design criteria for atomisation plants are dominated by the requirements of the powder market, product flexibility, high product quality for different alloy grades, minimum operating costs and safe operation.

To ensure the highest product quality at an acceptable cost inert gas recycling has become increasingly important for argon, and, as energy costs increase, it will also start to affect nitrogen.

Special features such as a redundant tundish system, pre-heating of the atomisation gas, the possibility to change between free fall and closed coupled atomisation systems, separation valves between powder tower and melt chamber and changeable atomisation towers for different alloy grades are tools which allow the powder manufacturer to handle a wide range of different alloy grades at the same quality level.

In order to keep the powder quality high after atomisation the down-stream handling of the powder will also become more important. This means that the powder should be protected at all times by inert gas to avoid any pick up of oxygen and moisture.

For materials with the highest requirements in terms of contamination, reactivity and melting, contact between the liquid melt and a ceramic lining have to be avoided. For these materials a ceramic-free melting and atomisation technique is required, such as with the EIGA process.

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