Process Metallurgy for High Performance Materials

Basic Research / Process Development / Semi Scale Production

This article describes the research and development capabilities in the field of vacuum metallurgy at Aachen University, Department of Process Metallurgy and Metal Recycling (IME). The fundamental methods needed to describe the process mechanism are presented as well as the existing semi-pilot equipment in order to experimentally validate the entire process technologically and economically. The equipment park is also open for partners to be used for semi-scale production, like sputter targets, ingots or powders.

Der Artikel beschreibt die Forschungs- und Entwicklungsmöglichleiten auf dem Gebiet der Vakuummetallurgie am Institut für Metallurgische Prozesstechnik und Metallrecycling (IME) der RWTH Aachen. Es werden die grundlegenden Methoden zur Darstellung von Prozessmechanismen erörtert, sowie das am Institut vorhandene Equipment vorgestellt, in dem die experimentelle Validierung der gesamten Prozesse hinsichtlich technologischer und ökonomischer Aspekte erfolgt und in dem Spezialprodukte wie Targets, Ingots oder Pulver auftragsbezogen hergestellt werden.

1 Introduction

Today’s demands on high performance metals and metallic materials can often be met only when using ultra pure raw materials, by employing processes avoiding oxygen and nitrogen pickup and application of high-end equipment serving productivity and economy. Their applications/customers line up from targets for physical vapour deposition (PVD)-coating, ingots for biomedical implants, getter granules/powders for electronics and gas purification up to semis for further metal forming treatments. The IME, the Department of Process Metallurgy and Metal Recycling of RWTH Aachen University undertakes development on the basis of its fundamental research group of integrated processes for the production of such high performance materials. Starting from master alloy manufacturing using extra clean aluminothermic reduction (ATR) methods or vacuum induction melting (VIM) the full range of well-established remelting methods like VIM, vacuum arc remelting (VAR), electron beam melting (EBM), electro slag remelting (ESR) can be combined and tested in both lab- and semi-pilot scale. A subsequent treatment like vacuum annealing, zone melting, mechanical machining, inert gas milling or hydriding/de-hydriding (HDH) powder synthesis can be finally integrated in order to supply tailor made products. Computerized process control and the use of most recent sensor technologies ensure the high precision of product composition, homogeneity and reproducibility. This article describes the IME’s present know-how and the possibilities for joint projects or material supply to research partners in universities and in the industry.

2 Basic Research

Understanding the thermochemical, kinetic and physical fundamentals of all process steps is essential for any attempt of process optimisation. Based on the knowledge of the reaction mechanism, process modelling is possible in order to simulate parameter changes and to investigate their impact on the product quality and process efficiency.

The variety of lab- and semi-pilot-scale equipment at the IME allows a subsequent validation of the modelled results and the adjustment of the models. Strong advantage derives from being part of the technical University of Aachen offering the possibility to easily integrate all scientific disciplines into our projects if needed. The following three chapters will give a short glimpse into our fundamental methods.

2.1 Thermochemical Modelling of Processes

One of the main fundamental activities of the IME is focused on the evaluation of thermochemical equlibria between liquid phases like molten metal and slag. Usually, also solid phases (e.g. refractory materials) and the furnace atmosphere are taken into account. The development of new metallurgical processes demands for accurate knowledge about the phase equilibria in composition or temperature ranges which often have not been examined before. Two different methods are used to generate models that allow the prediction of these equilibria and reduce the number of experiments to be conducted.

Mostly a new process does not only depend on thermochemical properties of the phases but also on kinetic and mechanical parameters. A statistical model gives a first general overview about the dependencies between targets and parameters. Usually, a semi-factorial experimental plan is used to reduce the number of tests. In promising ranges more tests are conducted to increase the significance of the results. In spite of this it is nearly impossible to receive accurate information about the process, since the model only consists of experimental values and systematic errors cannot be excluded.

In addition to the statistical model, a thermochemical model is generated. Based on the current state of the research in free enthalpies (Gibbs energies) of pure components and solution phases, the optimizer software FactSage® is used to calculate the thermochemical equilibria in the examined system by minimizing the total free enthalpy. Missing interaction values can be measured at the IME by differential scanning calorimeter (DSC) and differential thermal analysis (DTA) and are included into the FactSage® calculation. The model is optimized by back-coupling with experimental results. This model produces more accurate results than the statistical model, since the fundamental data are evaluated under laboratory conditions, but it is often more time-consuming.

Figure 1 shows an example of the experimental validation of a thermochemical model which was used to calculate the stability of CaO-crucibles against different metals as dilute components in the liquid state (normalized to activity 1).

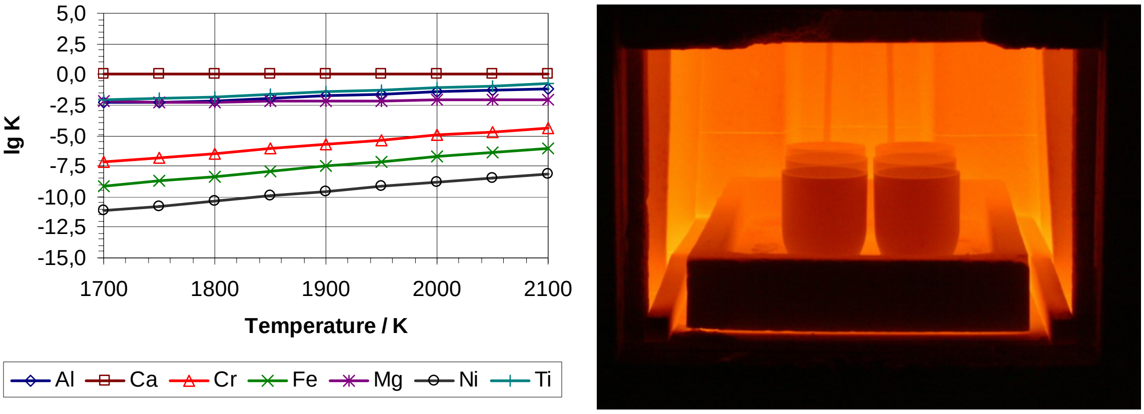

Figure 1: Evaluation of the stability of CaO-crucibles against different metals (left: model result, right: experiment)

2.2 Properties of liquid phases

A specialized lab is equipped with measurement devices for investigations of density, surface tension, wetting angle and viscosity of fluids. Especially the uncommon slag systems for ESR and ATR are not well explored and deserve closer attention. The applied resistance-heated furnaces allow a temperature up to 1700°C. In these furnaces a gas flow serves a defined protecting atmosphere. Platinum, corundum, molybdenum and steel crucibles and tools are used.

Density

For the density measurement the hydrostatic weighing method based on Archimedes' principle is used. A submerged tool is effected by the buoyancy of the volume of liquid it displaces. The result is that the tool weighs less in a liquid media than in air. It is necessary to know exactly the volume of the submerged tool, as well as its density dependence on the temperature. The density of the melt is calculated as

$$\rho_m - \rho_a = \frac{m_a - m_m}{V}$$

where ρ_m and ρ_a are densities of the melt and air, m_a and m_m the weights of the tool in air and in the melt and V is the volume of the tool at the corresponding temperature.

Surface tension

For the measurement of the surface tension a gas/liquid meniscus is produced at a capillary submerged in a liquid media. The pressure in the capillary is increased up to a bubble detachment from the orifice. The pressure in the capillary is continuously recorded. Using the maximum bubble pressure, the surface tension can be calculated from the following relation that is derived from the Laplace equation:

$$P_{max} = \frac{2\sigma}{r} + \rho g h$$

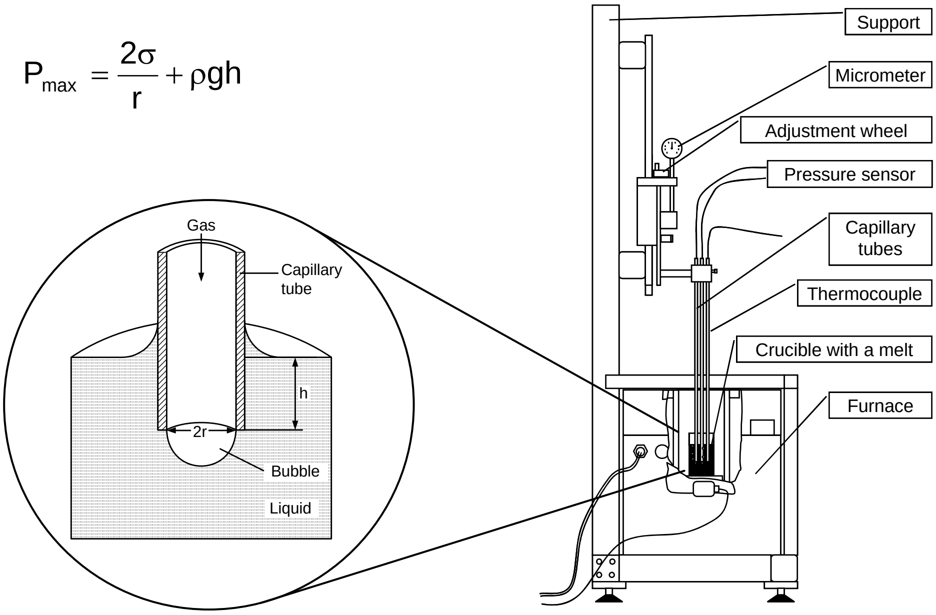

Figure 2: Measurement principle "maximum bubble pressure" and the associated device for surface tension data

Viscosity

The viscosity is measured using a plate oscillated by an input coil and a corresponding output signal from an output coil is measured. If the plate is immersed in a liquid sample, the oscillation of the probe changes its amplitude, phase, and frequency due to the change of the viscosity friction force. The method uses various measurement modes depending on the viscosity range. For viscosities up to 10 Pa·s the so called amplitude mode is used. In this mode the amplitude of the probe in air is compared with the amplitude in the fluid. The evaluation of viscosity results from a calibration curve, which is made by measuring fluids with known viscosities and densities (Figure 3). For this method the density of the fluid is necessary.

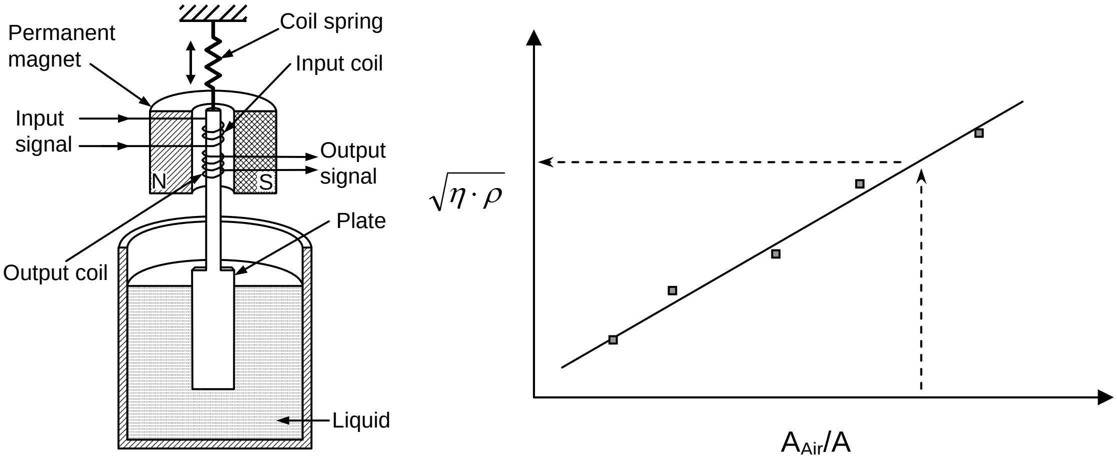

Figure 3: Measuring viscosity with oscillation method and the calibration curve for the evaluation

2.3 Kinetics and Reaction Mechanism

The optimisation of many processes can only be achieved, if beside the thermochemical equilibria also the kinetics and reaction mechanism are considered. In most cases the measurement of the kinetics is easy, since either a periodical sampling is conducted during the reaction or several samples are brought into the same reaction conditions for a different duration. The results are harmonised with kinetic rules, like the temperature dependence of reactions, impulse models or the kinetic gas theory. The quality of these results corresponds to the statistical model presented in chapter 2.1.

When the reaction rate is high or when the reaction only takes place in intrinsic reaction zones, in general the sampling is very difficult. When this is the case or when very accurate results are needed, the reaction mechanism must be understood. The whole reactor is then split into different reaction zones, which receive reaction products from other zones and transfer their products to third zones. The transfer rate is calculated by the formulas of heat, mass and impulse transfer.

Figure 4 shows exemplary the result of the measurement of the reaction progress during the aluminothermic reduction of titanium oxide. For the first 90 sec, the reaction front moves slowly downwards. 90 – 110 sec after ignition, the reaction rate increases significantly. Considering the mass transfer of the gaseous reaction products, it could be shown that in a first reaction phase, where the gaseous products move through the non-reacted mixture, the pressure in the reaction zone and thus the reaction rate is significantly lower than in the second reaction phase, where the gaseous products move through the melt.

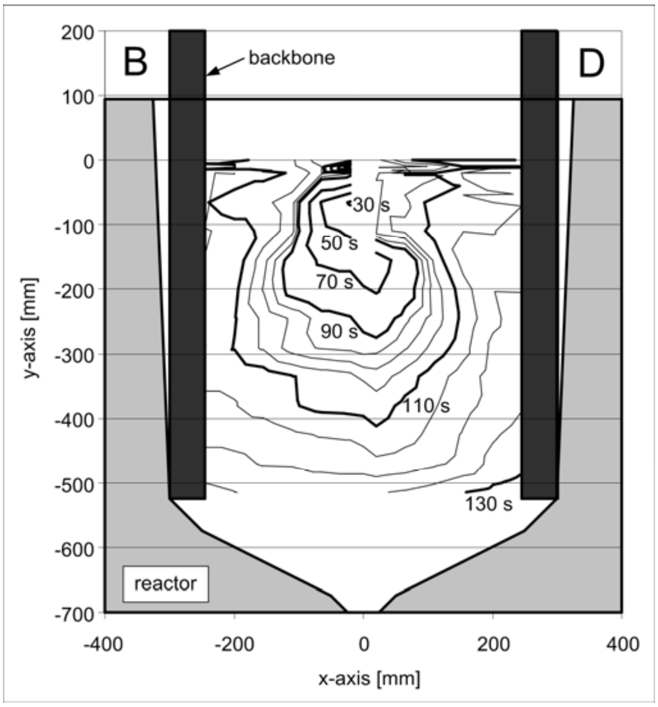

Figure 4: Reaction progress during aluminothermic reduction of titanium oxide

3 Process Development

As there is no „ideal“ process line allowing to produce all kinds of smart materials, the intelligent combination of industrially well-established process steps and of course promising innovations are essential to create suitable and efficient procedures. In the following chapters such key steps with their corresponding IME-equipment are shortly presented. Additionally, modifications and even new customized units can always be realized and tested.

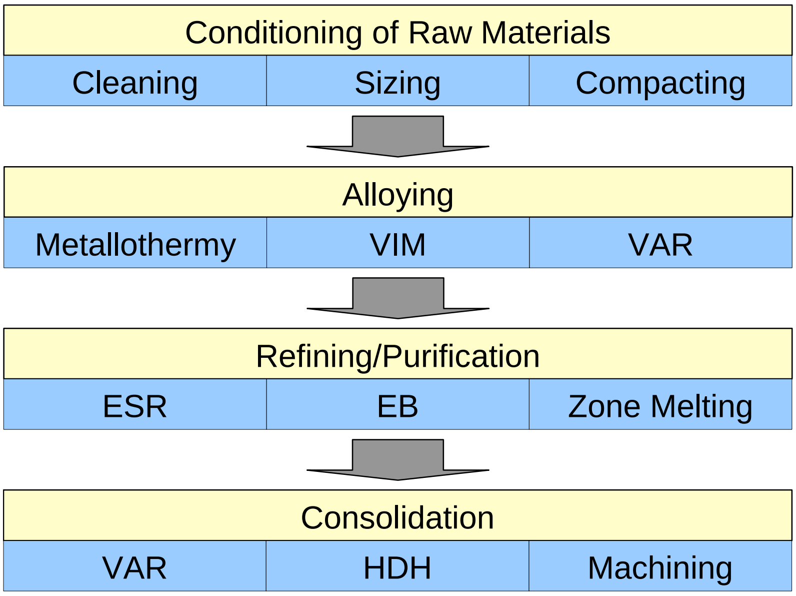

Figure 5: Process chain for high performance materials at the IME

3.1 Conditioning of Raw Materials

Many metallurgical processes have specific requirements for their raw material. A typical challenge of the synthesis of advanced materials is the preparation of homogeneous electrodes for the electro slag, vacuum arc or electron beam remelting made from sponge and/ or master-alloys. The recycling of turnings requires at least a compacting and delubrication process before the material can be remelted. If necessary, a laboratory rotary kiln can be used to volatilise lubricants. Alternatively, a washing process is possible.

At the IME a 500 t press was adapted to compact hexagonal blocks 100 mm x 365 mm from sponge, turnings or small scrap pieces. The blocks are either melted in a vacuum induction furnace or welded to primary electrodes and remelted in the (P)ESR-, VAR- or EB-furnace. Additionally the hydraulic press is used for compacting of powders for the production of sinter materials. Square green compacts of 200 mm length and 12 mm width can be achieved.

The maximum oil pressure of the hydraulic press amounts to 36 MPa, the distance between the dies is up to 1010 mm and the size of the dies is 420 mm x 420 mm. The piston can be moved with a constant velocity between 0 and 35 mm/min. At an adjustable maximum force, the press is switched off automatically. The force signal is indicated on an analogue 100, 200 or 500 t scale. Since the pressure is measured and supervised by mechanical transmission and there is no measurement of the piston velocity, future improvements will integrate an electronic control and automation system.

Figure 6: 100 mm die of the 500 t hydraulic press for the compacting of primary VAR and (P)ESR-electrodes from sponge or turnings

3.2 Metallothermic Reduction

A metallothermic reaction means the exothermic pyrometallurgical reduction of a metal oxide or halide by a metal with higher affinity to oxygen or to the halide respectively. Due to its high oxygen affinity, relatively low costs and acceptable boiling point aluminium is the most often used reduction metal. The composition of the reaction mixture determines the process temperature and, at the same time, the metal and slag temperature. At the IME a sophisticated thermochemical model, which is based on FactSage databases and experimental results, was developed to calculate the reaction mixture, considering incomplete reaction, evaporation and after-burning of components as well as heat losses by radiation and conduction.

The experimental laboratory equipment consists of two reactors with a reaction chamber volume of 32 l each. The usual amounts of the reaction mixture are 25 to 50 kg depending on the alloy. Both reactors can be used either for producing bulky material, which is crushed into pieces and can be refined by vacuum induction melting, or for an in-line casting of cylindrical ingots (ø 50 mm x 800 mm), which can be refined by electro slag remelting, vacuum arc remelting or electron beam remelting. A water-cooled copper mould is used for the in-line casting. Beside the laboratory reactors, a pilot-scale reactor with a total capacity of 200 l (160 to 300 kg) can be used for the production of bulky material or cylindrical ingots (ø113 mm x1340 mm). A ceramic mould is used for the in-line casting to reduce cooling stresses and the danger of cracks, especially relevant for brittle materials.

While the laboratory-scale reactors can be successfully used for the validation of the thermochemical model in terms of the alloy composition, the pilot reactor is needed to determine a mass and energy balance due to the reduced material and energy losses. A two-colour pyrometer can be connected to the moulds of all reactors for measuring the casting temperature of the alloy. Figure 7 shows a laboratory-scale reactor shortly after the reaction end.

Figure 7: 50 kg laboratory-scale aluminothermic reactor

3.3 Induction Melting

Induction melting (IM) is in most cases the first process step in metal and metal alloy making. Melting can occur in a protective inert gas atmosphere at standard pressure (IIM), under pressure (PIM) and, of course, in vacuum (VIM). The biggest process advantages of these methods are less melting losses due to eliminated atmospheric influences and the very quick homogenisation of the melt in respect to temperature and chemical composition because of the inductive stirring effect. An effective degassing and evaporation of unwanted volatile elements is possible under vacuum conditions. The IME operates five laboratory scale induction furnaces. The max. power supply is 40 kW by using a frequency of 10 kHz (middle frequency furnaces). Possible melt volumes are in the range of 100 ml up to 5 litres. Operation can occur under vacuum or protective gas atmosphere or also by using overpressure up to 0,3 MPa absolute for producing alloys containing elements with high vapour pressures (e.g. Mg or Li). Maximum melt temperature is 2000°C. Thus it is possible to test interactions between different melts and various refractory materials to choose the most reliable refractory material for producing high tech metals or to develop new kinds of alloys. A first up scaling step can be realised by using a 150 kW pilot scale furnace, commissioned in 2003. The maximum melt volume is 15 litres. Recharging of massive and/or bulk material and sampling of specimen during melting is possible. This permits after a quick spectrometric analysis an online adjustment of the chemical composition of the liquid alloy. Finally the melt can be tapped into conical or cylindrical water-cooled moulds made of steel or copper. Cylindrical ingots can easily be cast for further treatment as electrodes for VAR, ESR or EBM. Besides these options the whole crucible can be slowly moved out of the heating zone allowing a controlled continues solidification of the ingot, simulating DC casting. Optional the melt can be cast on water cooled rotating discs producing flakes for further powder production by HDH. An option of this VIM workshop is to allow a semi scale production of metallic materials, e.g. for structural and medical applications.

Figure 8: VIM workshop with the 150 kW pilot furnace in the background

3.4 Electro Slag Remelting

Electro slag remelting is a process for the production of nearly cylindrical ingots with high homogeneity, smooth surface and practically no inclusions above 5 µm. A slag bath is used as resistant which transforms electrical energy into heat and melts the primary electrode. The liquid metal is refined by the slag and solidifies in a water-cooled copper mould. High reactive materials like titanium can be remelted with oxygen-free slags under protective atmosphere (IESR, ChESR). Special applications (e.g. massive nitrogen enhancement) demand for higher atmospheric pressure (PESR).

The IME operates a combined PESR-ESR-furnace (Figure 9). While the ESR-furnace can be used for the production of 90, 160 and 200 mm-ingots, the PESR-furnace is restricted to 160 mm-ingots. The maximum size of the electrodes is ø110 mm x 1340 mm. The power capability (65 V ~, 4,2 kA) is actually increased to 80 V ~ and 6 kA, due to the higher demand of the oxygen-free slags. The control unit is upgraded and supplied with a new visualization software from ALD Vacuum Technologies GmbH as well. Thus it’s easier to transfer experimental data to production furnaces. The PESR-furnace can be operated in rough vacuum mode or in pressure mode up to 5 MPa. An inert gas cover system can be used to decrease the oxygen/ nitrogen partial pressure above the slag in the ESR-furnace.

Figure 9: Combined PESR and ESR furnace (left) and detail of the ESR crucible (right)

Current research projects are dealing with the desoxidation of titanium, titanium alloys and intermetallic titanium aluminides by use of active Ca-containing slags. Moreover the massive nitrogen enrichment of steel by pressure electro slag remelting is examined. Due to the high costs of primary titanium, a research project is currently carried out to recycle titanium scrap, like turnings and casting residues. The turnings are compacted and delubricated in a first step. Subsequently, a homogeneous ingot is produced from both materials by vacuum induction melting, alloying and casting into a water-cooled copper mould. The ingot is refined by electro slag remelting under protective atmosphere using an active Ca-containing slag. Finally, a VAR-step can be conducted to remove Ca- and F-impurities from the ingot.

3.5 Vacuum Arc Melting

Vacuum arc (re)melting can either be a first process step using compacted materials (e.g. titanium sponge with master alloys) as electrodes or as a subsequent process step using cylindrical ingots from VIM and/or ESR for remelting. The aims of the process are the further refining and the tuning of directional solidification. Due to the high quality of vacuum and the high arc temperature, the evaporation and/or dissociation of harmful compounds is possible. Oxides are floatated up to the top of the melting pool and transported to the crucible wall. Directional solidification is achieved by using a water-cooled copper mould. The IME will start up a new vacuum arc furnace in the 3rd quarter of 2006. The VAR is convenient for treating melting tests for research interests or also for small scale in-house production with a maximum batch size of 200 kg. Mould dimensions are 120, 160 and 200 mm in diameter, maximum electrode dimensions are 160 mm in diameter with a length of 1400 mm. The maximum power is 500 kW (7.5 kA, 70 V). Melting is also possible under protective gas atmosphere up to 0,1 MPa. This enables the minimization of evaporation losses of alloying elements and a positive influence on the solidification. By choosing the right mould a second and third remelting step is possible reducing large material losses by mechanical treatment. IME`s near future interests of research are measurements and benchmark tests of emissions (aerosol and particulate matter by means of vacuum pumps and processing emissions) of VAR applications by using particle sizer spectrometer and nanometer aerosol sampler. Another main research field will be the testing and optimizing of the implemented control equipment.

Figure 10: 200kg/200mm vacuum arc furnace to be delivered to the IME in 3rd quarter of 2006

3.6 Electron Beam Melting

Electron beam (re)melting (EBM) uses focused electron beams for melting of special metals and alloys. A well-established type of EBM is the so-called drip melting. A rotating electrode made of the material to be purified is (re)melted at the lower end thus the droplets pass the high vacuum atmosphere and collected in a molten pool. Due to the extreme local overheat of the melt and the very good vacuum conditions, degassing and evaporation of volatile unwanted compounds are the outstanding metallurgical effects of EBM. Directional solidification is achieved by using water-cooled copper crucibles and by moving the solidified material downwards. This is similar to the procedures in continuous casting. The IME operates such a drip melting EB furnace equipped with one electron beam gun for horizontal or vertical melting. The maximum power is 60 kW (2 A current and 30 kV acceleration voltage). The maximum electrode diameter is 100 mm with a maximum length of 350 mm. Remelted metal shapes can either be cylindrical ingots (max. ∅ 50 mm), slabs (100 x 350 mm) and even granulated materials. Centrifugal casting is also possible. An actual research field is about the dissolution kinetics of TiN and WC in liquid EB molten TiAl6V4.

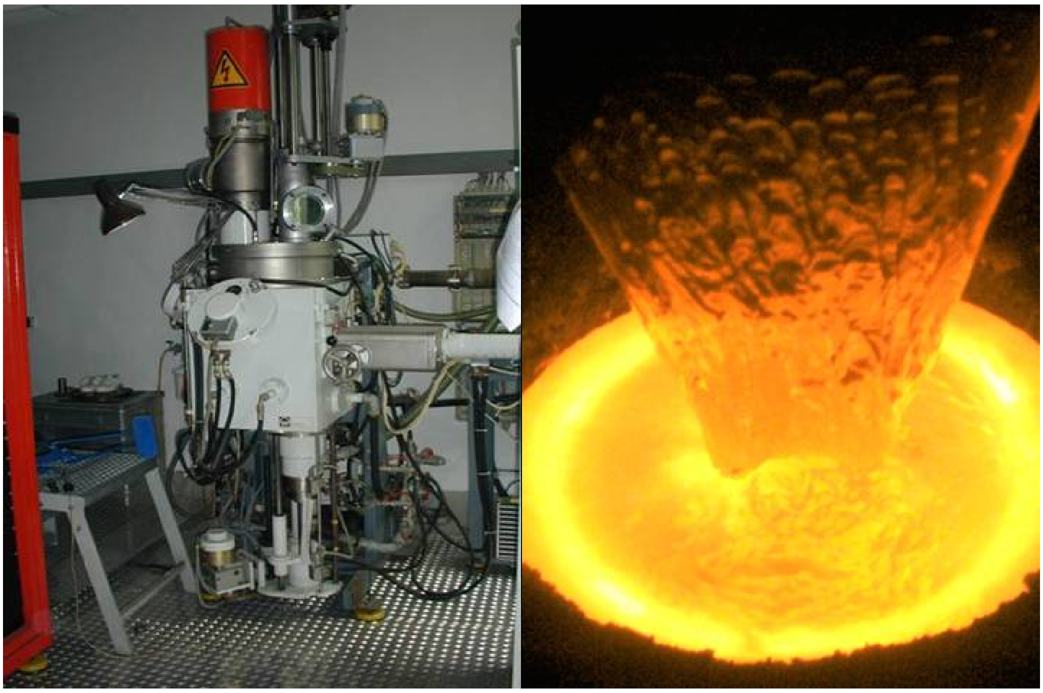

Figure 11: 60 kW single beam electron beam furnace (left) and drip melting electrode (right)

3.7 Zone melting

With the IME´s horizontal zone refining equipment high-purity aluminium < 99.9995 % Al was produced. During one pass, this device can purify 35 kg of aluminium. During the refining procedure, the aluminium ingot is placed in a graphite boat. The maximum ingot dimensions are 100 mm in diameter and 1500 mm in length. Such ingots can be produced in the semi-pilot vacuum induction furnace or supplied by the industry. The heating device has a maximal output power of 80 kVA. The induction coil is fed with a 10 kHz AC current.

Figure 12: 80 kVA zone refining device for ingot sizes up to 100 mm in diameter

This device can of course also be used for other metals and materials, like copper, silicon, iron, nickel, cobalt or aluminides and semi-conducters. The higher melting point of materials can be compensated by using ingots with smaller diameters and by employing graphite suszeptors.

3.8 HDH (metal powder production by hydrogen embrittlement)

The hydride-dehydride (or hydrogenation-dehydrogenation) method is used to produce powders based on rare metals/alloys, like titanium, zirkonium, tantalum or cerium. As raw materials sponge, billets, scrap or other forms of metal can be used. They are introduced in a sealed reactor. After evacuation, the container is heated and flushed with hydrogen, usually to some mbar overpressure. The hydrogen cracks/removes the oxygen layer and permeates the structure of the metal. The hydrogenation takes place during cooling below the 0,1 MPa-isotherm and is an exothermic reaction. Hydrogen is continuously absorbed in order to form an hydride at a constant pressure level. A slight system overpressure has to be controlled automatically. The product phase is a brittle metal hydride, like (Ti, Zr)Mn_2H_2 or Mg_2NiH_7, which is subsequently crushed or which even spontaneously splits into micrometer-sized particles. Depending on the specifications, the crushing/milling has to be carried out under cover gas. For the dehydrogenation, being obviously an endothermic process, the temperature has to be elevated significantly higher than the 1bar-isotherm and hydrogen leaves the metal lattice. This process can be accelerated by using vacuum pumps. The product at the end of the process is a nearly oxygen-free sinter-active metallic powder, usually showing sizes of 50 – 150 μm, and, of course, pure hydrogen gas.

The obtained powders may not be exposed to gases, like oxygen or nitrogen, since otherwise the pyrophoric product would react immediately, thus involving the risk of an explosion. For the final removal of the powder from the reactor, an inert gas has to be used. It is collected through a seal and can be led directly to downstream processes. A semi-continual production is possible. This method produces non-spherical metal powders at low costs.

Figure 13: Lab-scale-HDH (hydride-dehydride) furnace

4 Small Scale Production

The existing technology in Aachen allows the manufacturing of customer specified products in batch sizes from 1 kg up to 200 kg. Single melt options (VIM, VAR, ESR, EB), double melt options (e.g. VAR-VAR, VIM-VAR, VIM-ESR, VIM-EB, EB-EB) and triple melt options (e.g. VAR-VAR-VAR, VIM-ESR-VAR) are offered to synthesize almost all kinds of special metallic products. In order to show the vast variety, the following chapters present selected alloys and tailor-made shapes.

4.1 Master Alloys

One of the typical production processes of master alloys for steel, super-alloys and titanium consists of an aluminothermic reduction step, followed by a vacuum induction melting for the removal of slag inclusions. The alloy is cast into steel or water-cooled copper moulds and finally crushed into pieces. Due to the high costs of this procedure, several new alternatives are examined at the IME.

Figure 14 shows an aluminothermically reduced titanium aluminide master alloy, which was broken into pieces in a jaw crusher. Crushing is a well-working process to divide slag inclusions from the metal phase, since the material has significantly lower strength at the contact area between metal and slag inclusion. As a consequence, cracks grow primarily close to the inclusions. A final conditioning step by eddy current separation is used to divide both fractions. It is a precondition for this process that the aluminothermic reduction is well understood and the composition of the alloy can be adjusted precisely.

Figure 14: Aluminothermically reduced master alloy after crushing and eddy current separation

All high performance materials have very high standards concerning impurities, e.g. low oxygen and low aluminium contents of the master alloys for their synthesis. Since it is impossible to simultaneously reduce both impurities during the aluminothermic reduction, a refining step is needed. It could be shown, that remelting either crushed material or ingots in the electron beam furnace allows the removal of both impurities by evaporation of Al and Al_2O. A more economic process to produce master alloys with low oxygen and aluminium contents is the under-stoichiometric aluminothermic reduction followed by an electro slag desoxidation process with active Ca-containing slag. During electro slag remelting, high reactive or volatile elements can be alloyed by adding wire or granules.

4.2 Ni/Fe/Co-based superalloys

Superalloys on the basis of iron, nickel and cobalt contain minor elements, like Al, B, C, Cb Cr, Mn, Mo, Ta, Ti, W and Zr. They are used in the aerospace industry as well as in the engine and turbine production. They have outstanding high temperature properties and show high strength and corrosion resistance.

The conventional production route of superalloys starts in a vacuum induction furnace. The vacuum prevents the oxidation of base elements and therefore supports the exact meeting of specifications and also impedes the introduction of gases into the melt that would lead to porosities in the casted material. For all superalloys a remelting process is obligatory. ESR- or predominantly VAR furnaces are used as remelting aggregates. In the aerospace industry, up to three remelting steps are required. The remelting enhances the homogeneity and, thus, the properties of the alloys. Remaining impurities and especially solid inclusions are removed and the grain size and morphology is adjusted. At the IME, the whole process chain of the superalloy production can be reproduced in pilot-scale, that means in scales of up to 200 kg per batch.

Figure 15: Invar-ingot after VIM-cast and ESR-refining

4.3 Titanium and titanium alloys

After commissioning a new vacuum arc furnace in summer 2006, the IME will be able to perform the complete production of titanium and titanium alloys starting from titanium sponge and alloying element oxides in an 80 – 100 kg-scale. In case of producing TiAl6V4, the high purity V_2O_3 and V_2O_5 is aluminothermically reduced to a low oxygen containing V60Al40 master alloy, as described above. The master alloy is crushed, conditioned and pressed with titanium sponge to compacts. The blocks are welded to primary electrodes, which are double- or triple-melted in the VAR. Titanium alloy powder can be produced by hydration and dehydration (HDH) under vacuum.

Figure 16: VIM cast (Ti, Zr)Mn_2 hydrogen storage alloy before and after hydrogen embrittlement

4.4 Intermetallic Phases

Intermetallic phases like titanium aluminide or nickel aluminide are manufactured for applications as sputter targets or engine blades. TiAl can be produced through the conventional VAR-process route. For this purpose, titanium sponge, aluminium granules and alloying elements are mixed and compacted. By tungsten inert gas welding, these compacts are welded together to a primary VAR-electrode, which is double- or triple-melted in the VAR and finally mechanically shaped.

Nickel aluminide can be produced in the pilot-scale induction furnace VSG 100. For this purpose, the primary materials nickel and aluminium are charged into a suitable crucible and molten under vacuum. For controlling the specifications samples are taken under vacuum, analysed in the IME-lab within minutes by arc-spectroscopy and further material can be charged to meet the required composition. Furthermore, the temperature of the melt can be measured before casting under vacuum.

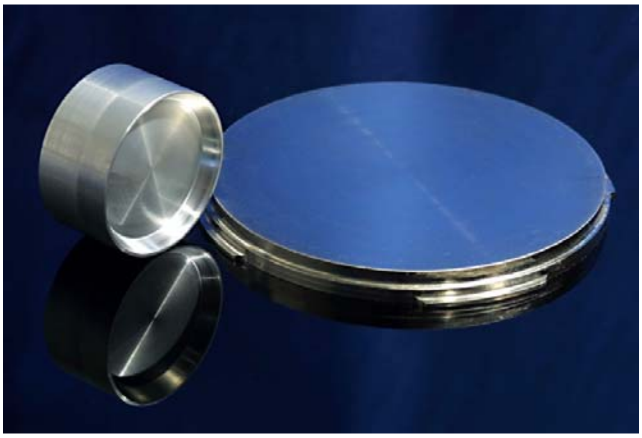

Figure 17: Typical sputter targets made from intermetallics

4.5 Refractory Metals

At the IME, refractory metals like niobium, tantalum, vanadium or molybdenum and their alloys can be produced by the aluminothermic reduction of the corresponding oxide. Equation 1 expresses the reaction in the ATR exemplary from niobium:

$$3\ Nb_2O_5 + 16\ Al = 6\ NbAl + 5\ Al_2O_3 \qquad (1)$$

For this reaction, an excess of aluminium is used, producing a niobium-aluminium alloy. The obtained unrefined metal with ingot diameters of 100 mm is refined in the electron beam furnace in order to evaporate the aluminium and to produce low-oxygen, carbon-free niobium. By multistage remelting the concentration of the impurities can be decreased into the ppm-area. Table 1 shows the maximum concentration of the impurities after the refining in an EB-furnace

Table 1: Concentration of impurities after remelting in an EB furnace

Impurity	C	O	H	N	Si	Fe
max. conc. in ppm	25	50	1	30	10	40

The diameter of the ingots after the remelting in the EB-furnace amounts to 100 mm. In an optional process step, multiple ingots from the EB can be remelted in the vacuum arc furnace to a bigger ingot with a diameter of 160 mm.

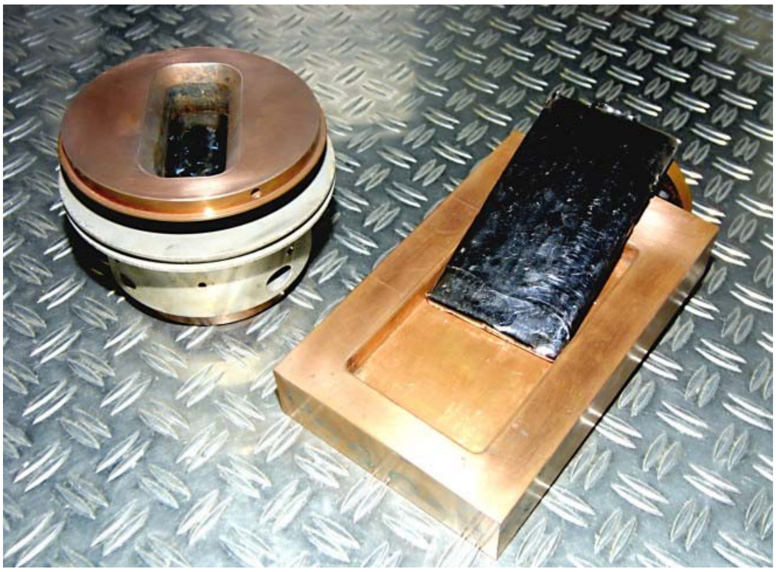

Figure 18: Electron beam melted niobium