

Latest Remelt Technics With Inductive Heating Under Vacuum / Protective Gas Atmosphere

Introduction

In modern laboratories of the metal producing and metal working industries as well as in the field of alloy research, foundry, metallurgy and nuclear technology, emission and x-ray fluorescence spectrometers are used since many years. These instruments are for the most part fully automatic and computerized - they are able to analyze in routine up to 50 elements in one sample.

In most of the metal producing industries the sample is taken directly via samplers from the melt and delivered the quickest possible way e.g. via pneumatic post, to the laboratory.

In the metal working industries the sample is a finished part and not suitable for analysing with the spectrometer due to its outer dimensions. Nevertheless the sample has to be controlled in order to meet the specifications of the customer or supplier. In this case chips, drillings or small pieces are taken from the finished part and remelted to a suitable sample so that the spectral analysis can be done.

The same is applicable for metal scrap. Nonferrous heavy metals, pig iron with a high carbon as well as silicon, ferro alloys, super-alloys up to precious metals now are treated only according to this process.

Additionally to the remelt technique the principle of the induction melting has a lot of advantages. It is quick, precise, nearly without losses, easy to handle, reproducible and cost saving.

When using a technique permitting to evacuate the sample chamber then to add protective gas under over-pressure there remain nearly no more wishes regarding losses, oxides, as well as homogeneity. Modern control technics with microprocessor control help to exclude human errors of these units.

The working principle

Induction melting units work according to the energy transmission principle of transformers. There is also a primary and a secondary coil. While the electromagnetic flux of a transformer is made through a laminator iron core as coupling element between the primary and secondary side, the energy transmission with induction heating is directly achieved through the electromagnetic field. After having switched-on the high frequent magnetic field, a secondary current occurs on the surface of the metal. Due to the different characteristics of the materials, this current is converted into heating. These eddy currents heat the metal up to the melting point at sufficient energy supply - overheating up to any casting temperature is no problem.

The eddy currents in the melt produce a so-called bath movement and with these electro-motoric powers a good mixing of the melted sample is guaranteed.

It is well known that Oxygen, Nitrogen etc. influence the sample negatively respectively make a proper melting impossible.

Even flushing the crucible and sample with protective gas was not sufficient. This is especially valid for the loss of carbon, manganese or components of the alloy which due to the vaporpressure lead to loss - at the desired melting respectively casting temperature. This ends in complicated calibration works and bad reproducibility and can be avoided by the combined use of vacuum and protective gas.

To avoid oxidation, a vacuum of 50 to 1 mbar would be sufficient. This, however, has the disadvantage that some elements at high casting temperature and also when there are large surfaces (chips, powder) are evaporating very strong and fast.

Optimum remelt conditions can be obtained when filling the preevacuated sample chamber with protective gas before reaching the casting temperature. It is an advantage to feed the gas into the sample chamber with a higher pressure at compared to the ambient pressure.

A relevant control system of the unit should exclude misoperation especially in the sector of time control, vacuum, gas flushing of high frequency power supply as well as of melting and soaking times.

Construction and function

Each induction remelting unit consists in its basic construction of a powerful high frequency generator transmitting the produced high frequency to a working coil which fits the melting crucible. Due to the required HF-powers to be able to remelt safely metal with a high melting temperature e.g. Platinum, tube generators with a high-frequency of 3 to 6 kW and frequencies of 0,8 to 1,8 mc have proved most suitable. Semi-conductor generators are not used due to the matching difficulties and to the low circular impedance as well as the fault susceptible expenditure of components. Water-cooling of the complete high frequency part is necessary with these high induction coil currents.

After expiration of the melting process the liquid sample solidifies and then can be taken out as a disc for surface treatment for the spectral analysis. Also the melting crucible can be taken out of the coil with pliers and then be pored in a mould to a disc. With that the work for machining is reduced.

Since several years the centrifugal casting proved best for the practical use of remelt technics. The vacuum pressure casting which is a way leads to more sealing problems as well as more additional handling. The centrifugal casting is done in practise as follows: The sample material is put into the ceramic crucible which is put into the crucible holder and then covered with the protective gas cover or the vacuum/protective gas container is closed through the cover with a viewing glass. During the melting process Argon flows with a flow rate of 0,5 - 5 l/min. into the crucible. The air is displaced in only a few seconds and so the oxidation of the melt is reduced. With modern vacuum/protective gas units the chamber is evacuated to an adjustable vacuum level and after a fixed time filled with protective gas under over-pressure. The entire casting arm is constructed as a well balanced system turning around its own axis.

The sample shape (mould) is inserted in this way that a ceramic spout can be put between the crucible and the mould to assume a pass of the casting stream with a good mechanical connection and almost lossfree casting. The casting arm with the ceramic crucible is positioned directly above the induction coil. The coil is lifted manually or through a servo-motor until it encloses the ceramic crucible.

The melting process starts immediately after switching on the high frequency. It takes approx. 30 to 90 seconds to reach the casting temperature depending on kind of material. After having finished the melting process, the coil moves down to its original position. When making different melts the micro-processor control of the unit can be adjusted to a certain holding time for cooling respectively stabilization of the melt. After this period, the casting process starts automatically with electronically limited spin time. By the centrifugal effect of the casting machine at approx. 500 rpm the molten metal is pressed into the mould. The adjustable casting torque of modern units guarantees the optimum casting of different material quantities, especially of metal having a very different specific weight. They solidify very quickly to a homogenous analysing sample (e.g. white structure). The whole procedure including the handling for operation of the unit lasts max. 2 minutes.

Material and sample

The sample material consists mainly of chips, pieces, powder, wire, drillings, wastes, scrap metals etc. from steel, GS, GG, GGG, GTW, GTS, aluminium, zinc, bronze, copper, nickel, titanium and other alloys up to precious metals. The material quantity and kind of crucible are chosen depending on the shape of the sample to be melted. The remelt technique allows to use quantities between 5 to 50 g; bigger remelt units which are mainly used for production of standards even allow a quantity up to 1 kg relating to the specific weight of Fe. Material with a low specific weight have a smaller quantity. They are limited by the volume of the ceramic crucible for others through the power of the HF-generator or of the desired melting time.

In cases where chips, powder or loose material is remelted, it is necessary to prepare in advance a mould suitable to the crucible. This is to limit the sample surface and with that the induction current to avoid overload of the high frequency generator. The other point is to avoid difficulties of falling or adhering on the upper crucible wall of chips. Shape and size of the chips don't have any importance. For production of the samples a pressure of 30 to 100 kp/mm² is sufficient. This process is similar when using powder samples. Here the whole sample may be thrown out of the crucible when switching-on the high frequency because of the eddy current.

A further risk is the blow of the protective gas stream or the suction of the vacuum pump. Also the coupling of bad conductive materials can be improved in many cases through compacting and/or pressing-in with a metal having better conductivity.

For spectral chemical analysis with X-ray fluorescence and spart emission spectrometers the remelt sample has the shape of a disc having a diameter of approx. 10 to 60 mm. This corresponds to a quantity of approx. 10 to 80 g Fe. Usual standard samples have a diameter of 35 to 40 mm and 5 mm thickness.

The mould consists of the outer base part, and an inner casting part which can be separated in two parts and also serves for fitting on the casting arm. With newer moulds, a separate disc can be put into the outer base part to avoid destruction of the mould in case the sample is casted too hot and, therefore, fuse to the mould.

Due to the mechanical and thermal stability, a zircona-copper alloy is used for making the copper moulds.

Of course there are some applications where moulds are made of high density electro-graphite are used especially in the nonferrous field. In any case with these moulds, pasting or fusing is avoided. Also in such cases the heat conductivity and with that quick solidification is reached satisfactory. Sometimes also ceramic moulds which were used only one time are taken after the dewaxing procedure to produce samples of super-alloys or tension test bars. Of course one can also simulate the casting behaviour respective to the change of casting materials of all alloys. To complete this we also want to mention moulds made of graphite monolithic lining material respectively rare earths.

Ceramic crucibles

While it was relatively simple to find a suitable material for the thermic charging of the mould, the search for a suitable crucible material still leads to problems.

The experience shows that different crucible materials are needed to be able to use the unit universally. This problem has perfectly been solved in cooperation with applicants of different fields. In the meantime there are several manufacturers world-wide producing most suitable materials for remelting crucibles. Patents of manufacturers of remelt units even on the sector of crucible geometry fulfil every request.

The four main demands for a crucible are: Several meltings in one crucible (factor of cost); no impurities of melt through the crucible material (contamination problems); good temperature resistance to thermal shocks (working speed); different crucible volumes (flexibility). Today's crucibles are based on SiO₂, Al₂O₃, ZrO₂ or TiO₂ material. Due to that they have a short time temperature resistance up to 2000°C which is sufficient even for high-alloyed, high melting materials.

Practical experience

Through the induction heating followed by automatic casting process under vacuum/protective gas, you have samples which - under normal conditions this means according to the normal analyzing regulations for steel samples - can be analyzed very well with emission and X-ray spectrometers. When watching for example the distribution of sulfur in a remelted sample of machined steel, it may be observed that because of the quick cooling, the sulfur is more uniformly distributed than in other samples. The determination of concentration of carbon and manganese is made e.g. with calibration curves which are established new for the remelting samples as with these elements a low melting loss has been shown which is due to the Hydrogen contents in the sample or due to the remaining air in the mould. However, with modern induction remelting units where the casting arm is made as a vacuum/protective gas chamber, for some elements an improvement by the factor 3 has been achieved compared with normal remelting units flushed with protective gas. This effect disappears nearly complete when adding deoxidants like aluminium or zirconium with a content of 0,1 to 0,2 % in the shape of thin foil or strips.

Determination of carbon in pig iron and steel samples with spectrometric analyzing systems is only then guaranteed when the sample solidifies white. This means when carbon is uniformly distributed. Out of experience the white solidification is for sure reached with modern remelting procedures.

Today many ferro-alloys are remelted by inductive heating e.g. ferro-vanadium, ferro-manganese, ferro-chrome, ferro-phosphor, ferro-niobium, ferro-molybdenum, ferro-zircon and ferro-silicium.

This can be obtained when mixing the ferro-alloys with high pure iron. The mixing rate is by experience in the range of 1:2 to 1:5 e.g. 1 part of sample to 3 parts of mixing substance e.g. high pure iron. For remelting of some samples e.g. ferro-vanadium the adding of high pure iron is not sufficient. Only after having added 3 to 5 % nickel the melt gets the required quality. Beside the relative high temperatures for ferro-melts (1500-1800°C) which presuppose an extreme temperature resistance and low solubility of the ceramic crucible, the crucible material is heavily chemically attacked as the crucible reacts very strong and fast with the melt at these high temperatures. After purposeful tests suitable crucibles for these applications could be manufactured. There are no limits for dilution of samples respectively for addition in order to get an optimum remelt result. Nevertheless, should there appear a problem then so-called lining would help i.e. the inner wall of the crucibles is treated e.g. a thin layer of chrome oxide, cobalt-oxide or boron nitride mixed with a binder and shortly fired in a lab furnace, coats the surface and diffusion speed is reduced.

It goes without saying that today graphite, silicium carbide crucibles etc. are used when heating non-ferrous metals. These crucibles are formed this way that they can be put into the standard ceramic crucible which have the effect as an insulating container.

Control

The fact is that the analysis of samples meanwhile is done in nearly most cases computer-controlled and fully automatic. The next step was to equip also a high-quality sample preparation unit with a micro-processor, including protective gas flushing or vacuum/protective gas operation. With this integrated small-computer all safety and operating functions can safely be controlled and displayed. The operation of the function such as vacuum, gas, high-frequency, holding time, casting time, cooling water flow, overload, excess temperature and safety interlocks can easily be managed under the elimination of human errors. In case a number of equal samples

shall be remelted (melting/casting time) the process can be fully automated through an optical infrared measuring system which - due to the problems of different emission factors - has been calibrated relatively i.e. in percent against a black radiator. This means that the sample material has to be put into the crucible, the unit to be started and the finished remelted sample to be taken out.

Summary

Remelting through inductive heating with a vacuum/protective gas unit gives the metallurgical laboratory in research and production individual samples and the control with a number of advantages to the usual procedures for sample preparation. The utility of this technique offers considerable advantages in the reproducibility, low losses as well as a high working speed. This has been confirmed by many users of these units in various application fields from platinum to iron.

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normal steel	C 0,063	SO 0,31	Mn 1,46	P 0,015	S 0,001	Al 0,04
Remelting (without additional elements)						
normal	0,051	0,22	1,09	0,016	0,001	0,01
vacuum	0,049	0,27	1,28	0,015	0,001	0,02
vacuum/gas	0,052	0,28	1,39	0,016	0,001	0,02
Remelting (+ 1 g C)						
normal	---	0,28	1,36	0,013	0,001	0,02
vacuum	---	0,32	1,43	0,013	0,001	0,04
vacuum/gas	---	0,31	1,45	0,014	0,001	0,04

Case 1: Due to Hydrogen content in the sample, melting losses can not be totally avoided. Losses are very low when using C (- 0,01%) - but when using MN and Al they can clearly be seen. MN melting loss is constant when remelting under vacuum atmosphere and can be calibrated.

Case 2: When adding Carbon, the Hydrogen is binded that no correction with Mn or Al is necessary when remelting under vacuum or vacuum/gas atmosphere.

Annexures

- picture 1 induction principle
- picture 2 bath movement in the melt
- picture 3 casting arm for normal version with protective
gas flushing with crucibles and moulds
- picture 4 vacuum-casting arm with ceramic one-time-use mould
- picture 5 useful accessories
- picture 6 calibration curve for the determination of carbon in
remelted ferromanganese samples with high-pure iron (1 : 3)
- picture 7 picture of the remelt unit Lifumat 3,3 µP VAC
- picture 8 micro-processor control of the unit with control panel