

Controls and quality demands in the jewellery investment casting process

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The investment casting process, extensively used for the production of jewels, is made up of different phases that, for their intrinsic typology, are deeply influenced by many chemical, physical as well as metallurgical factors. Even though investment casting has been used for decades, it is absolutely not properly optimised, also in consequence of the difficulty in deeply understanding, and therefore controlling, all the different phases of the process. It is therefore evident that different, and unfortunately many, kind of defects may appear in precious art-crafts.

The identification of the defect nature represents a decisive step in order to determine its cause; the comprehension of such an aspect is, consequently, the first important action to prevent the defect re-appearance and, then, for the optimisation of the entire process.

The present paper presents a general overview of casting defects and quality demands in jewellery and takes into exam some possible “non conformities” that can arise in the production of jewels. The defective samples have been examined throughout metallographic observations together with the use of SEM coupled with EDS analysis.

Parole chiave: metalli preziosi, difetti, microscopia elettronica

INTRODUCTION

Even though it is almost an utopia to produce “perfect” objects through investment casting, a successful process is, in jewellery, tremendously important to obtain a standard “defect-free” products. For the complexity of the process and for the numerous different steps that are part of it, many difficulties may arise during the production.

Therefore, when a defect is observed, rapid actions should be taken in order to eliminate the causes that were at its origin. However this can be done only if the nature of the defect is known and the cause is well understood.

Sometimes the simple observation with a lens at 10 magnification is not even sufficient to classify the defect (i.e: what kind of gas porosity?), and more detailed analysis are needed; in some cases the only possible solution is to use a SEM together with EDS in order to identify the nature of the problem.

Once having realised the nature of the defect, the way to the identification of its cause is traced; the following step should be at this point the searching for the proper actions to be taken in order to avoid the defect re-appearance.

Different and extensive studies have already been carried out in this field [1 - 5]; this paper aims to shortly summarise possible defects in casting, their causes and general solutions, as well as to be a further contribution for producers to realise the importance of controls and quality demands in this specific sector.

Table 1 correlates, in general terms, some of the most common possible defects and their main causes.

Defects	Possible causes
Gas porosity	Decomposition of the refractory, calcium sulphate and/or oxides in recycles, oxidising atmosphere, oxides in molten metal (including zinc oxides), carbon (from crucible)
Shrinkage porosity	Shrinking during solidification, misruns, incorrect filling
Rough surface	SO ₂ formation
Sandblast surfaces and slobbers	Excess of water, investment failure
Watermarks	Excess of water
Inclusions	Refractory decomposition, alloying elements, slags from recycles,
Bubbles	Incorrect refractory preparation cycle
Cracked surface	Incorrect flask burn out cycle
Crystallisation	Wrong cooling rates

Table 1: Defects in investment casting and possible causes

Tabella 1: Difetti del processo di microfusione e possibili cause

DEFECTS DERIVING FROM THE ALLOY COMPOSITION

The defects that can be introduced during the alloy preparation phase are not belonging exclusively to the field of investment casting for jewellery. However, it is particularly in this area that the effects deriving from non properly designed and produced alloys can determine different types of defects/non conformities in the final products.

In this paragraph some cases deriving from “external” contaminations or non properly optimised processes in preparing the alloys, at least in terms of alloying elements, are examined. The analysis of the defects that can be deriving from “dirty” or un-slagged metals is reported in the following chapters.

Figure 1 shows the surface of an object with evident defects. A more detailed analysis, carried out in different areas of the

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Memoria presentata al Convegno:
Cera persa e investment casting: innovazione arte e tecnologia,
Alessandria, 9 ottobre 2003

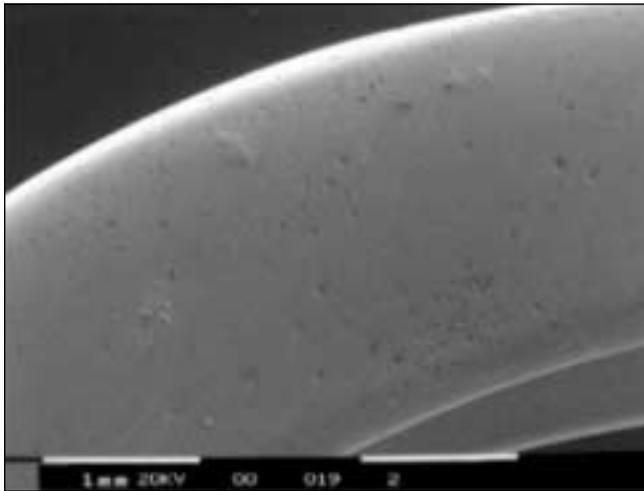


Figure 1: SEM image of a defective object at the final stage of production

Figura 1: Immagine SEM di un manufatto finito difettoso

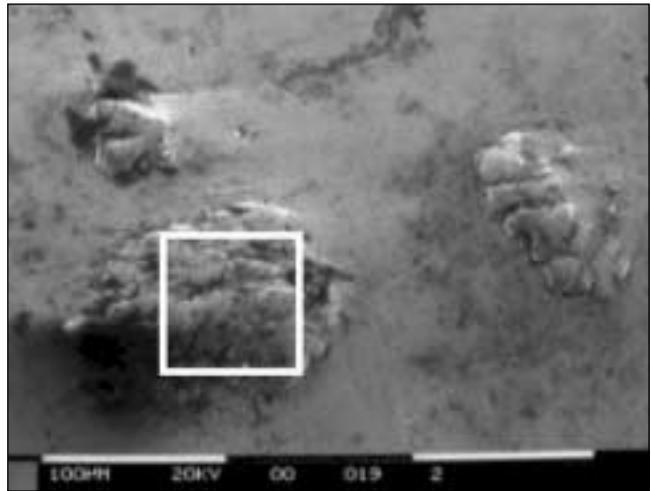


Figure 3: Detail of defective area

Figura 3: Dettaglio di area difettosa

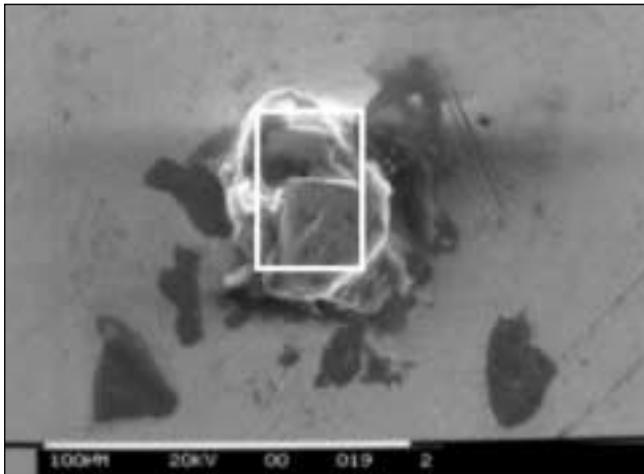


Figure 2: Detail of defective area

Figura 2: Dettaglio di area difettosa

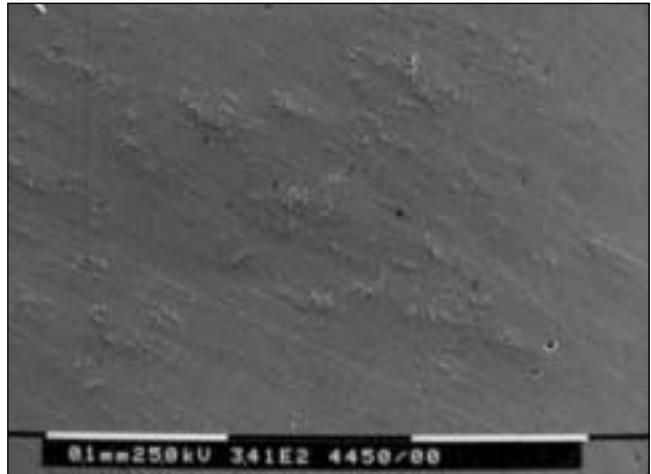


Figure 4: Defects observed at SEM at low magnification

Figura 4: Difettosità osservata al SEM a bassi ingrandimenti

sample, underlines the presence of different elements, as shown in Figures 2 and 3.

The EDS analysis, performed in the marked areas, put into evidence the presence of osmium (Fig. 2) and iridium (Fig. 3), which are not reported to be present in the base alloy preparation phase. Their presence is therefore to be lead to the refining step of the starting pure gold.

Microstructures reported in Figures 4 and 5 show some defects deriving from the presence of iridium, in this case used as grain refiner in the production process.

In this case, some segregated areas rich in Ir are observed. It can be supposed that the origin of the defect is due to an improper mixing during the preparation of the alloy.

GAS POROSITY

In this category different types of defects, deriving from different gases, can be included. In the following lines the most frequent cases are examined.

Porosity deriving from SO₂

The surface undergoing this kind of defect shows many small and rounded porosity, which are damaging the quality of the surface and of the inner layers; in any case the mechanical properties of the object are generally not deeply influenced, at least

not to a level of causing catastrophic failures, differently from what happens in case of other porosities.

Two different cases can be identified:

1) In case the porosities are located at the surface or in its close surroundings, the cause should be identified in the decomposition of the refractory, which may take place during casting and even before, during the burn-out cycle.

In fact calcium sulphate starts to decompose at different temperatures, depending on the operating conditions, shortly summarised as follows:

- between 1200 e 1240°C in case it is heated up on its own;
- between 1000 e 1100°C in presence of cristobalite;
- between 750 e 900°C in presence of carbon.

When the decomposition of calcium sulphate takes place, the object of reaction reacts with copper and/or silver, giving raise to the formation of sulphides.

As an example, the reaction with copper can be summarised as follows:



Sulphides then react with oxides or with oxygen (copper oxide can be contained in the AgCu alloy, while silver can contain relevant quantities of oxygen), leading to the formation of sulphur dioxide SO₂ that, generally, is trapped in the molten alloy, causing at the end porosities.

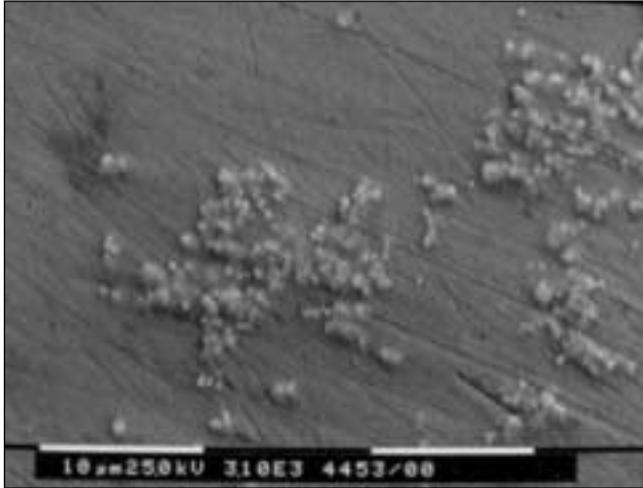


Figure 5: Detail of figure 4 at higher magnification
Figura 5: Dettaglio della figura 4 a maggiori ingrandimenti

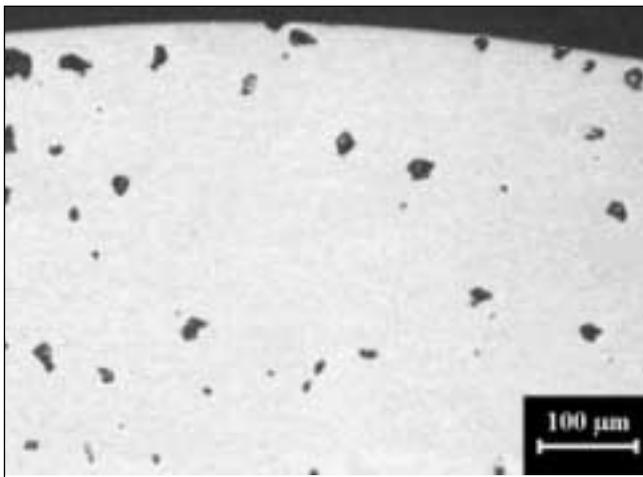


Figure 6: Typical gas porosity from SO₂
Figura 6: Tipica porosità da gas da SO₂

The efficiency of de-waxing is therefore of great importance, as wax may not be totally eliminated, with the absorption of a fraction of it by the refractory. If that's the case, the following step is then the formation of carbon based residuum. As seen before, carbon brings to a faster decomposition of the refractory and this causes mainly a decrease of its mechanical properties and, at the end, porosity in the object. Therefore the main reason for keeping high temperatures during burn out is to fully eliminate carbon residuals.

2) In case porosities are present along the transverse section, they may generally be related to recycled metals re-introduced into the process. In fact they might be containing traces of refractory deriving from previous castings. The mechanism leading to the formation of SO₂ is similar to the one previously described.

The surface affected with these types of porosities can undergo surface finishing; however this operation may further increase surface porosity in case the defect is located just near the surface itself. It is therefore necessary to prevent the sulphur dioxide formation, reducing the oxides present into the alloy and treating scraps to be re-used.

Porosity deriving from oxygen from the decomposition of CuO
This defect includes two different types of surface porosity: small rounded and bigger ones having a more irregular shape.

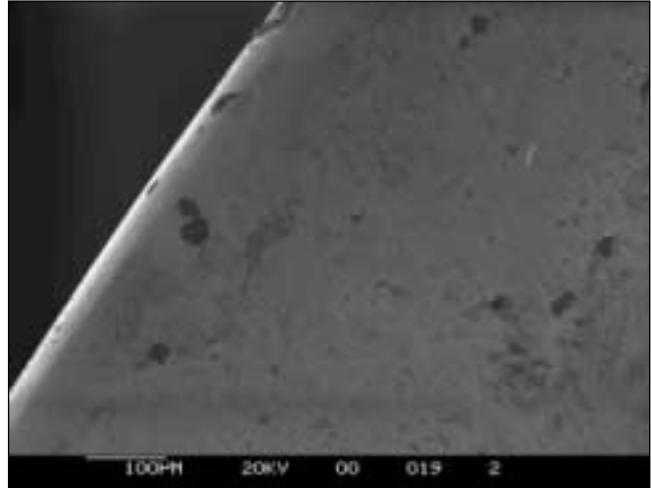


Figure 7: Rounded SO₂ porosity located at the surface of the sample
Figura 7: Porosità tondeggianti da SO₂ situate nella zona superficiale del campione

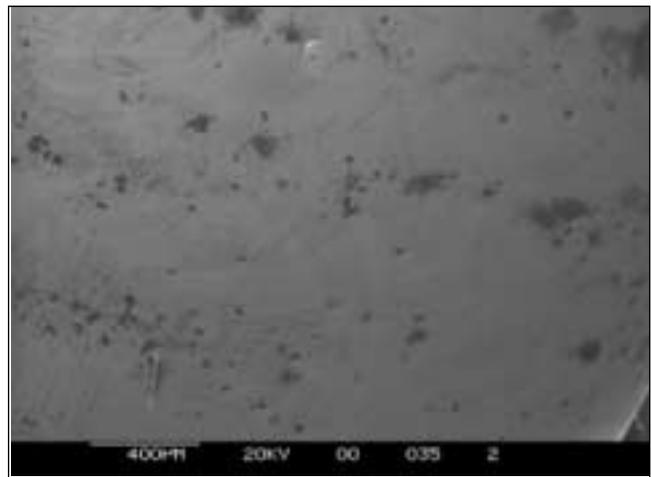


Figure 8: Rounded pores located near the surface
Fig. 8: Porosità arrotondate collocate in prossimità della superficie

They are mainly due to the presence of copper oxide inclusions, generally introduced through the re-use of scraps during the preparation of granules.

The aspect of this defect can be confused with shrinkage porosities; anyway its different nature can be related to the presence of CuO. Copper oxide, whose stability is strictly dependent on temperature, modifies its equilibrium with copper during solidification with the release of oxygen, generating porosities. It is therefore necessary to use precautions to minimise this problem, such as casting under non oxidising atmosphere, limiting to 50% max the quantity of recycles and, most of all, take special care of the "cleanness" of these latter, with the final aim of reducing the presence of oxides.

Porosity deriving from CO

The study of different types of porosity has recently lead to the identification of carbon monoxide as the cause of some defects which cannot be included in any other kind of either gas or shrinkage porosity.

The formation of carbon monoxide may take place when the following conditions are present:

- Starting material containing oxides
 - Casting atmosphere containing oxygen of water vapour.
- The oxides, the oxygen or the water vapour can combine with carbon coming from the crucibles, generally made up of graphite, thus creating CO determining porosity. However, in

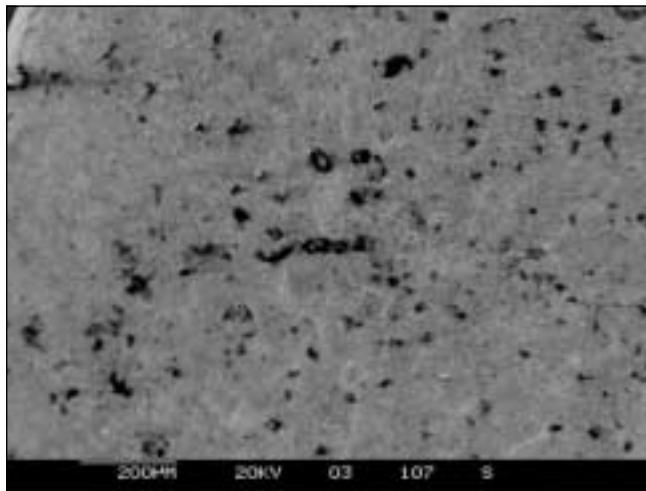


Figure 9: Example of porosity deriving from the presence of CuO ; bigger pores are evident in the centre of microstructure

Figura 9: Esempio di porosità da CuO ; pori di dimensioni maggiori sono evidenti nella zona centrale

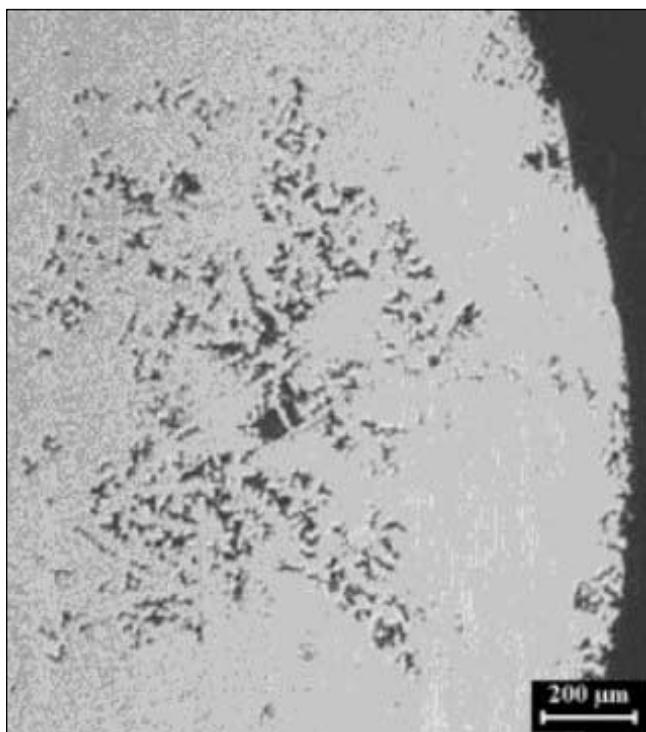


Figure 10: Example of porosity related to shrinkage

Figura 10: Tipica porosità da ritiro

case the metal is kept in its molten state for a longer period, CO is allowed to leave the surface of the crucible more easily. An increase in solidification time can therefore contribute to eliminate this problem: the solidification can also be delayed through an increase of the flask temperature: the refractory retains more energy and metal stays in its molten state longer. This “trick” may however lead to the formation of SO_2 ; however a general reduction of porosity is observed and this underlines the predominant role had by porosity related to CO . As the refractory form filling, also the removal of carbon monoxide depends on the efficiency of the filling system: in case fillers show a certain resistance to the flowing molten metal it is useful to increase the casting pressure, thus making easier the removal of trapped gas and reducing the risk of having pores. Generally the pores dimension is related to that of the grains of cast object: a fine-grained structure does not entirely re-

duce the defect, but at list determines a homogeneous distribution of small pores.

SHRINKAGE PORES

This type of porosity is caused by the volume reduction due to the transition from liquid to solid state. In the investment casting of small objects (as most of jewels are) the process of solidification is more complicated than for bigger ones. In fact, while in this latter case the solidification starts from the outside to the inside and from the bottom to the top, in the former one it is more complicated in consequence of the small dimensions and of the complexity of shapes, deeply influencing the heat transfer process.

Generally solidification occurs first at the borders, where the molten metal transfers the heat to the die walls and quickly proceeds in the thinner part of the object, where the heat transfer is faster. Cluster of dendrites start forming in the molten metal; while the solidification proceeds, voids among dendrites form as consequence of the solidification shrinkage without the possibility of being filled, since no molten metal is available. Moreover the dendritic ramification would create an obstacle to the flux of metal. A partial solution to the problem is to introduce additional molten metal through the filling gates.

Figure 10 shows typical shrinkage porosity.

Differently from what happens for gas porosity, in this case a deep influence on mechanical properties is present: a “spongy” and porous structure near a critical area of the finished product locally reduces the mechanical resistance, with the real possibility of having ruptures even with low applied loads. A proper change of the burn out and of casting temperatures may partially contribute to avoid the premature solidification of additional molten metal, provided to compensate shrinkage.

The dendritic structure can determine another type of defect, different from the previously mentioned porosity: rough surface.

It can be mainly seen on gates and on objects having bigger dimensions, that's to say those interested with higher heat transfers and therefore with an increase of temperature detrimental for the refractory. In this case the cause is to be searched, once again, in the calcium sulphate decomposition and in the consequent SO_2 formation.

During solidification crystallites keep growing in a dendritic form originating networks; molten metal localises into the interdendritic areas, until sulphur dioxide determines its movement far from the surface. The result is a dendritic framework at the surface.

The defect can be prevented decreasing the casting temperature, to reduce the refractory decomposition; this can however cause an incomplete filling and therefore an increase of casting pressure should be performed.

The addition of small quantities of zinc may be favourable for the reduction of this type of defect.

SANDBLAST SURFACES AND SLOBBERS

These two defects are located in the surface areas and are both deriving from the refractory weakening that generally deteriorates, being unable to sustain the casting pressure and abrasiveness.

The object surface is then rough (figure 11), while at the borders micro-cracks form in the refractory; molten metal penetrates in these areas and determines irregular shaped slobbers (figure 12).

Refractory failures can be caused by:

- Poor quality of the refractory stock. The quality and the resi-

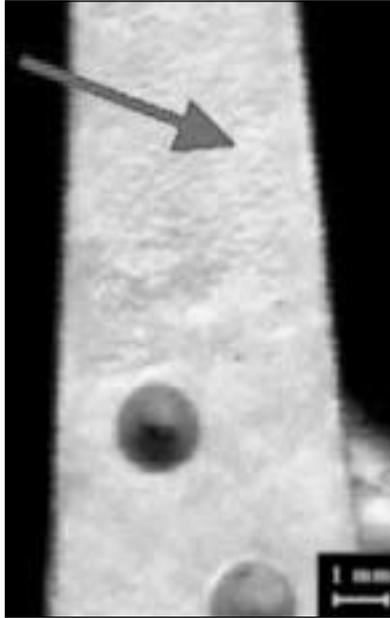


Figure 11: The arrow indicates a sandblast area located at the top of the object

Figura 11: La freccia indica un'area interessata da superficie sabbiosa

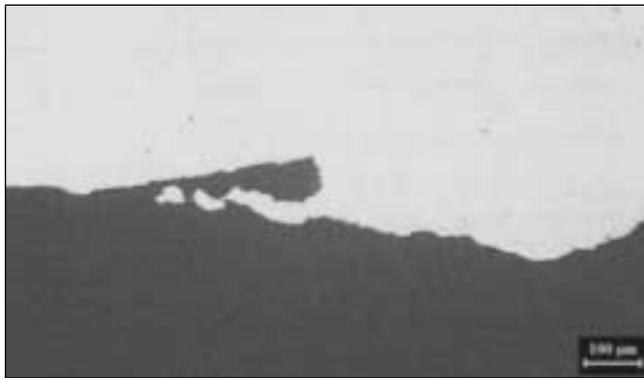


Figure 12: Typical example of slobber

Figura 12: Tipico esempio di sbavatura

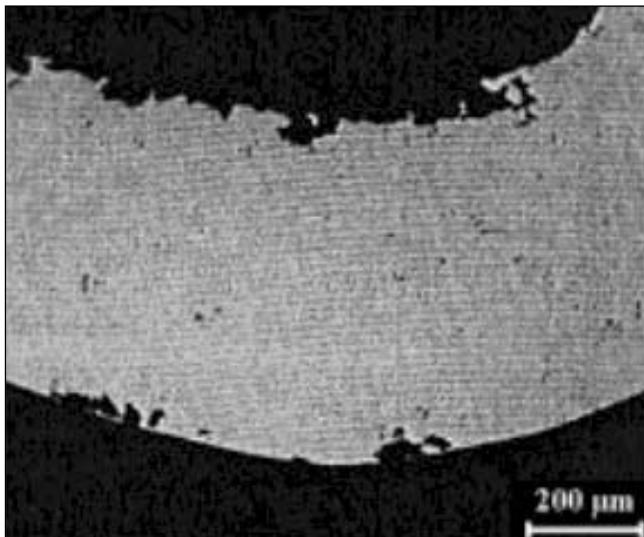


Figure 13: Porosity deriving from refractory inclusions

Figura 13: Porosità derivante da inclusioni di refrattario

stance of the whole system are determined by calcium sulphate, since it acts as a binder for the refractory. It is tremendously important to keep CaSO_4 stored in a dry environment.

- Excessive quantity of water in the investment preparation; this can be the cause for an inadequate bonding action of calcium sulphate towards cristobalite.
- Too fast heating cycles during flask burn out. Part of the wa-

ter is eliminated between 190 and 200°C. The process has to be slow enough; otherwise the whole resistance is reduced.

- Eventual decomposition of calcium sulphate.

The degree of extension of the defect is related to the pressure of the filling of molten metal into the flask; the higher the pressure, the higher the possibilities of having poor surfaces. In centrifugal castings this defect is rather frequent, since the high rotational speed generally determines a high casting pressure. It has to be kept in mind that, during the process, higher pressures on walls are located at the bottom of the flask, hence causing in this area the majority of defects.

WATERMARKS

This type of defect results in different tracks present on the surface of the product generally oriented in parallel to the flask. They can be eliminated through the final object polishing in case of flat and simple shapes, while in case of more complex surfaces there is no solution but recycling.

Watermarks originate when an excess of water is present in the investment mix. Since this latter is a mix of water, binder and refractory, mixing has to be carried out for the proper time. The same problem can take place when too short working times are applied and/or the whole system is not moved in the minutes preceding the hardening: water and solid may separate and part of the liquid stays on the wax surface. Marks left on the investment remain also after de-waxing and are reproduced on the final object.

It is then necessary to weigh water and calcium sulphate on the correct proportions but, most of all, mixing under the proper conditions. Again CaSO_4 should be stored in a dry and conditioned environments.

INCLUSIONS

Different types of inclusion can be present in jewels obtained through investment castings. The typical metallic inclusions (i.e: zinc oxides) are here just mentioned and elsewhere deeply investigated [6]. The attention in this paper is oriented to non-metallic inclusions.

Object can be porous with evident cavities randomly distributed at the surface and/or with inclusions located just close to the surface. Also this type of defect can be related to the refractory decomposition, even though the main cause is not the sulphur dioxide formation: inclusions are constituted by refractory particles, while inside cavities there used to be the same particles, which were removed during surface finishing operations.

Figure 13 shows a typical example of cavities/ poor surfaces deriving from the presence of inclusions.

During castings, the melting metal can partially remove part of the investment, especially in presence of turbulent flow. Non metallic particles are then trapped into the object as inclusions. The causes for the refractory crumbling have to be searched in the poor quality of investment powder or generally in some mistakes during the preparation of the tree, with the positioning of edges in critical areas as, for example, in the central gate joining points.

Generally this type of defect does not decrease the mechanical properties of jewels.

A similar defect, although deriving from a totally different cause is shown in the following figure 14. In this case, as before, it is present a non-metallic inclusion, as it can be easily seen in the analysis spectrum but differently from before, the defect composition cannot be related to a refractory decomposition. Alumina can derive, in general, from operations carried out before the preparation of the alloy as, for example, from previous finishing operation on the alloy then recycled. It could

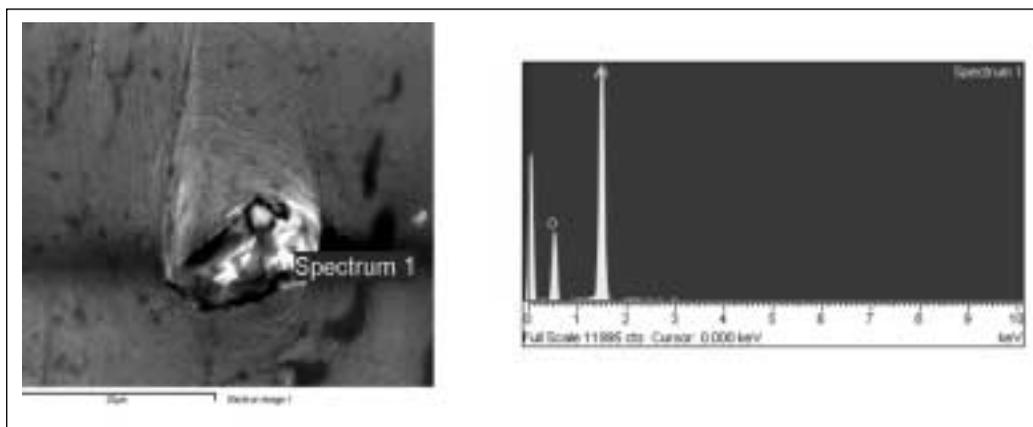


Figure 14: Non metallic alumina based inclusion

Figura 14: Inclusione non metallica, avente composizione riconducibile ad allumina

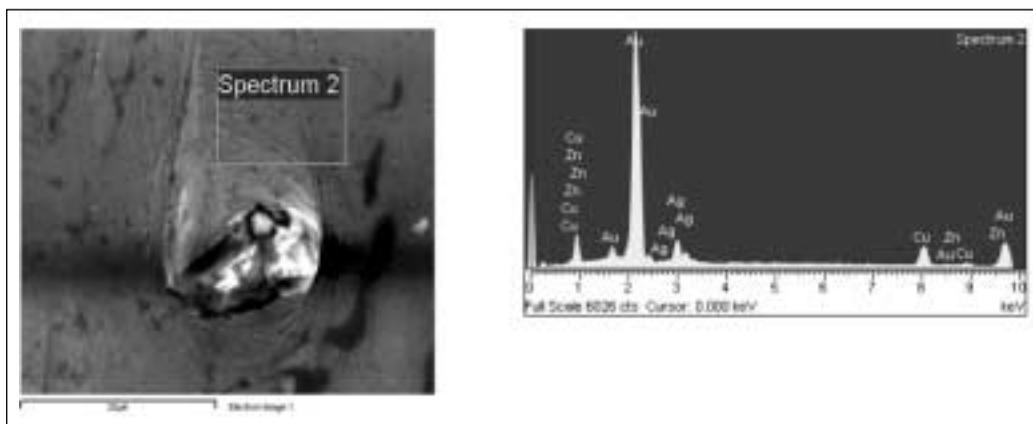


Figure 15: Analysis of the alloy located close to the non-metallic inclusion

Figura 15: Analisi di una porzione di lega in prossimità dell'inclusione non metallica

have been brought to surface during finishing processes. The second analysis (Fig. 15), performed in the surroundings of the previous one, underlines that the problem is deriving from an external problem and not from an incorrect preparation of the alloy.

CONCLUSIONS

The present paper shows some of the defects that can arise during the production using investment casting. For the high number of variables present in the process, it is rather difficult to obtain a standard set of actions/procedures to be done, valid in any case and for any considered alloy. However the always growing demand of higher quality product coupled with the request of cost reduction (and the necessity of more finishing processes as consequence, for example, of a surface defect, can be considered an additional cost) is tracing the road towards a deeper research of the optimised working conditions. In this field modern instruments for research can play a

very important role, for the identification of the cause of the defect and for the following actions to be taken to avoid the defect re-appearance.

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ABSTRACT

CONTROLLI ED ESIGENZE DI QUALITÀ NEL PROCESSO DI MICROFUSIONE PER LA GIOIELLERIA

KEYWORDS:
precious metals, defects, SEM

Il processo di microfusione a cera persa, ampiamente utilizzato per la produzione di manufatti preziosi, è costituito da una serie di fasi che, per la loro tipologia, risultano influenzate da molteplici fattori di natura chimica, fisica e metallurgica.

Pur essendo utilizzata da decenni, questa tecnica produttiva non è però ancora del tutto ottimizzata, anche in conseguenza dell'oggettiva difficoltà di capire a fondo, e quindi con-

trollare al meglio, tutte le fasi del processo. Risulta quindi evidente come si possa avere, nei pezzi, la comparsa di difettosità di vario genere.

L'identificazione della natura del difetto rappresenta una parte fondamentale per determinarne la causa; la comprensione di tale aspetto è, di conseguenza, il primo passo per impedirne la ricomparsa e, quindi, per l'ottimizzazione dell'intero processo produttivo.

Il presente articolo traccia un quadro generale dei difetti di colata e delle esigenze di qualità nell'industria orafa, prendendo in esame alcuni difetti tipici della produzione di oggetti microfusi su cui è stato eseguito un dettagliato studio metallografico e di composizione anche mediante analisi con microscopia elettronica abbinata ad EDS.