

**“BUT I’VE ALWAYS DONE IT THIS WAY.”**

**TECHNICAL SUPPORT - IT MAKES A DIFFERENCE.**

by

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**Abstract.**

Manufacturing jewellers have their own preferred methods for producing jewellery and have used these successfully for years. Occasionally things don't work out as expected and it's very easy to blame the bullion or semi-fabricated materials supplier and their product. This may not necessarily be the case and problems can originate from the jewellers' process and conditions. In these cases a good after-sales technical service from the supplier is invaluable to give quick and economic resolutions to problems. This paper highlights several examples where the failure mechanism is not that first suspected. The paper will demonstrate that good technical support can save time, money and give an advantage over competitors as well as giving the jeweller a better understanding of the materials and processes used.

**Introduction.**

The jewellery industry in both the UK and US has over recent years become increasingly technology based. There are manufacturers who prefer traditional techniques however many have moved away from the “artisan” mentality favouring light engineering techniques - progression tooling, multi-parameter control in investment casting, CAD/CAM etc. As a result specifications on supplied semi-fabricated materials have increased dramatically. This has led to an increase in the requirement for technical support in alloy selection, property determination and particularly trouble-shooting where materials problems are encountered. To this end a bullion or semi-fabricated materials supplier should be expected to provide technical back-up as part of their after-sales package giving the customer a full range of services. If you purchase a new TV and it breaks when you try to use it, you don't throw it away and accept scrap value without first requesting an investigation to determine what has gone wrong and why. Was it your fault or an inherent defect in the particular TV you purchased? In the majority of industries this type of after sales service is expected as a matter of course. Should the jewellery industry be expected to supply a similar service? Jewellery manufacturers produce the items that are sold to the public but are also customers and as such should expect the same range of services.

I attended the Santa Fe Symposium for the first time in 1997 and was impressed with the range of papers presented and topics discussed. Papers were written with no obvious commercial bias and were purely for the purpose of supplying information to the jeweller. This paper has been written using the same emphasis and will demonstrate that the use of a suppliers' Technical Services Department is an invaluable tool to the jewellery manufacturer. It may be used to trouble-shoot and determine origins and resolutions to problems that may otherwise have proven expensive to resolve. Manufacturers cannot be expected to have a knowledge of all the analytical techniques available and as such may be unaware of their existence and potential.

Since 1990 I have been a member of the Technical Services Department of the largest semi-fabricated jewellery materials supplier in the UK. The department is responsible for supplying a technical after-sales service to customers and consists of three sub-areas:

- Metallurgical - responsible for in-house production trouble-shooting, alloy development and customer technical enquiries and rejections.
- Production Engineering - responsible for the identification, purchase and installation of plant and equipment for the company. This area is also responsible for plant and equipment related trouble-shooting.
- Analytical Chemistry - responsible for in-house assay services to ensure all product despatched reaches minimum assay requirements. This area is also responsible for compositional analysis and impurity determination.

The Metallurgical Department is responsible for the customer services discussed in this paper. It is a service offered to customers that is strictly objective in nature, the aim being to resolve problems to a satisfactory conclusion and not to deflect blame away from the company. It can be considered a free technical consultancy service. Advice is commonly sought on alloy and process selection, the majority of cases being dealt with over the telephone however site visits occur when necessary.

With a broad experience in customer problem solving one aspect becomes apparent - the majority of customer problems are related to their processes and conditions and are not inherent in the metal supplied. To this end the paper highlights six case studies to demonstrate this. In all cases the manufacturer believed the supplied metal to be at fault - superficial examination would tend to confirm this however more in-depth analysis revealed this not to be the case. These studies are not



intended to belittle or humble the manufacturers in question but to demonstrate that use of the Technical Services Department resulted in the rapid identification of problem origins and any corrective actions required. Problem solving by manufacturers is generally based on surface analysis - either by eye, loop or using a low powered stereo microscope - and experience. The latter quite correctly stands for a great deal but is no substitution for accepted analytical techniques such as the metallurgical microsection and electron microscope. When involved with investigations and site visits a great deal can be learnt from customer experience however it is clear there is still an acute technical ignorance of the materials and processes used due to a lack of basic technical expertise.

Outlined below are the techniques generally available for an investigation:

- Physical and mechanical property determination of alloys.
- Metallurgical analysis of failed workpieces by microsection. This enables the evaluation of structure, failure modes, failure mechanisms and also the identification of porosity, inclusions, oxides or other phases present.
- Further analysis by a range of other microscopic techniques including electron microscopy.
- Phase, inclusion and impurity determination by microprobe analysis.
- Semi-quantitative X-Ray fluorescence analysis for the identification of compositions and impurities.
- A range of further analytical chemistry techniques to determine compositions and impurity levels.



Before deciding which of these techniques to use a description of the problem and sample examination is required. For one-off problems a sample may be submitted through the Sales Office and a telephone discussion sufficient to allow analysis to begin. All samples are submitted through the Sales Office and logged into a rejection tracking system to ensure a record of what alloy and how much has been returned by whom. All relevant order numbers are also included on this document to allow analysis of production records to take place if necessary. For more involved problems a site visit by a member of Technical Services is usually advisable and samples may be taken at this stage.

Further information is necessary before analysis can take place:

- What is the metal - i.e. alloy, form and order requirements stated?
- What is the problem?
- What operations have been performed on the metal after receipt by the customer?
- Where in the process is the problem being identified?
- Is this a one-off problem or has this occurred before? Is it uncommon or frequent?
- Does the problem occur with alloys purchased from other suppliers or just ours?
- Is there any as-supplied metal available to confirm conformance to order requirements?

Armed with this information the required analysis may be carried out and the following questions answered:

- Has the customer stated the correct order requirements for the intended process?
- Was the metal supplied to the stated order requirements?
- If so where in the process is the problem occurring?
- What is the origin of the problem?
- What is the mode and mechanism of failure?
- What is the resolution to the problem to prevent recurrence?

Once the nature of the problem and the ideal corrective actions have been determined these may be discussed with the customer. It is possible a compromise may be required - for example if a customer has chosen a particular alloy for colour purposes but the alloy requires quenching after annealing and the customer has no quench facilities, an alternative alloy may be necessary resulting in a compromise in colour. A report is produced, initially for in-house records however if a copy is requested by the customer one can be supplied - this is generally the case. In the majority of cases the customer is extremely appreciative that someone has taken time to give technical assistance.

It must be noted that analysis and reports are never supplied on a competitors metal - this is considered extremely unprofessional. Opinion may be given in some cases however this will only apply when assurance has been given that it will not be used at any time for compensation claims on another supplier.

The requests received by the department for advice and analysis are wide ranging therefore the case studies chosen are all - bar one - based loosely on surface related problems.

**Surface contamination of a hand raised 18ct ladies handbag body.**

The first case study was submitted by a customer who employs a workforce of skilled craftsmen who manufacture high value often one-off made to order items typically from 18ct and 22ct alloys. Each craftsman is responsible for manufacturing the job from commission to finish and as such is skilled in the majority of precious metal fabrication processes, the workpieces often being hand formed from sheet and wire. This particular problem involved the appearance of cracks in the raised corners of one half of an 18ct ladies handbag. The customer made comments concerning a similar problem experienced when manufacturing a lampshade from an 18ct yellow alloy after soldering several large sheets together and raising over a former. The craftsmen involved did not believe the supplied sheet was annealed to the required standard resulting in a loss of ductility and subsequent failure due to overworking.

Historically the 18ct yellow alloy used by this customer has been 18ct HB which has a 2N composition of 75% Au, 16% Ag, 9% Cu. This is predominantly the favoured 18ct yellow alloy type used in the UK market as opposed to the 3N type - 75% Au, 12.5% Ag, 12.5% Cu - preferred in Europe. This alloy measures 125HV in the annealed condition however the customer had recently requested an alloy of similar colour but with a lower annealed hardness and increased ductility. The alternative alloy supplied was 18ct MBG which has a composition 78% Au, 15% Ag, 6.7% Cu, 0.3% Zn and retains the preferred 2N colour. The alloy is closer to 19ct however when considering the intended market the slight increase in caratage does not have an impact on profitability. There are other 18ct alloys available with increased ductility to 18ct HB and MBG however these do not have the desired colour qualities. Figure (1) shows the work hardening curves of the two alloys and demonstrates that up to 60% cold work the 18ct MBG has a lower work hardening rate.

The items produced by this customer are not always items of jewellery and as such may be physically large. The use of 2N type alloys has the advantage that the Au-Cu ratio places the composition outside the order/disorder transformation that effects 18ct alloys of higher copper



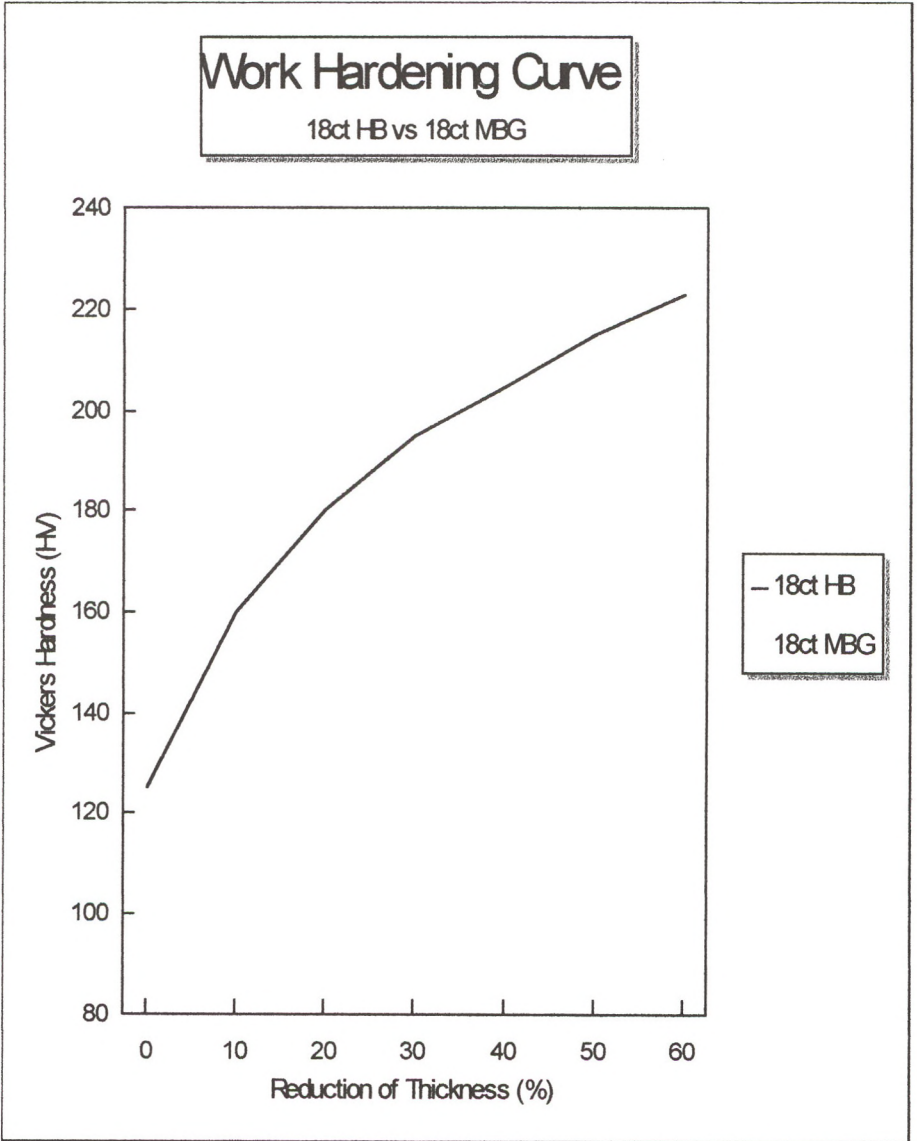


Figure 1. Work hardening curves of 18ct HB vs 18ct MBG.

content. Quenching may be used if an ageing treatment is required to harden finished items however it is not necessary for maximum ductility to be achieved - there is little or no difference between the elongation values for the quenched and air cooled states. 18ct HB may be softened further by a longer annealing cycle at higher temperatures however over annealing may occur resulting in grain growth, loss of strength and ductility. 18ct MBG was designed to match the 18ct 2N colour but with improved properties for hand working - hardness and elongation. It can be moderately age hardened but to a lesser degree than 18ct HB. It is suited to the requirements of this customer.

As previously noted the suggestion had been made that the supplied sheet was not annealed to specification resulting in failure due to over-working. There was no as-supplied sheet available to test conformance to order requirements however internal sales-order-processing systems enabled the order number to be tracked to a cast bar number and a final inspection report. This confirmed the sheet was within the order specification of 105HV maximum when tested at the final inspection stage prior to despatch to the customer. Initial examination of the workpiece revealed brittle failure had occurred with inter-granular cracking present throughout the failure area (figure 2). This type of failure is untypical of overworking and usually associated with failure due to the presence of an impurity. Microsectional analysis confirmed overworking was not the failure mechanism. There were extensive grain boundary failures present (figure 3); examination at higher magnifications revealed the presence of a grey grain boundary phase (figure 4). Analysis by X-ray fluorescence and atomic absorption spectroscopy revealed the presence of 200ppm lead in the bulk sample. It is well documented that lead embrittles gold alloys by forming a brittle low melting point grain boundary phase  $Au^2Pb$  (1,2,3,4). Sources quote different "safe" levels of lead in carat gold alloys however the majority agree that 200ppm will be detrimental to an alloy and seriously restrict further cold working operations. This usually applies to bulk levels in cast, rolled or drawn product. In this case the impurity has been introduced externally at local level and as such concentrations will be



Figure 2. Surface of failed 18ct MBG workpiece. Mag x 30.

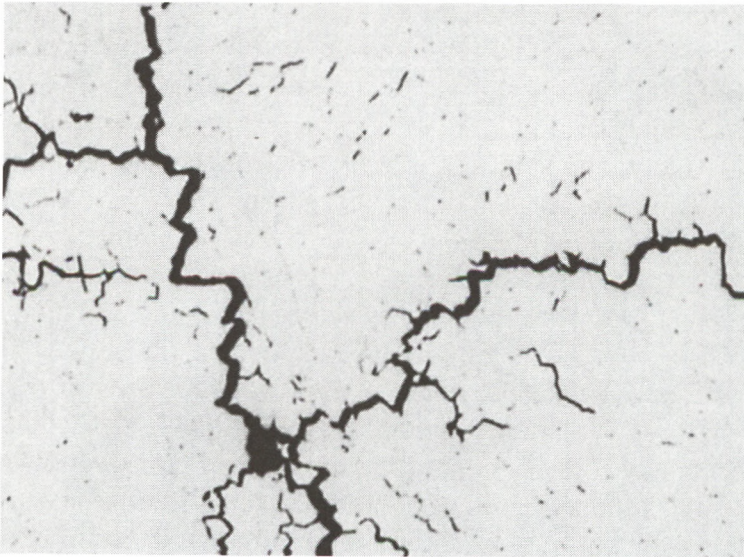


Figure 3. Inter-granular cracking in 18ct MBG. Mag x 80



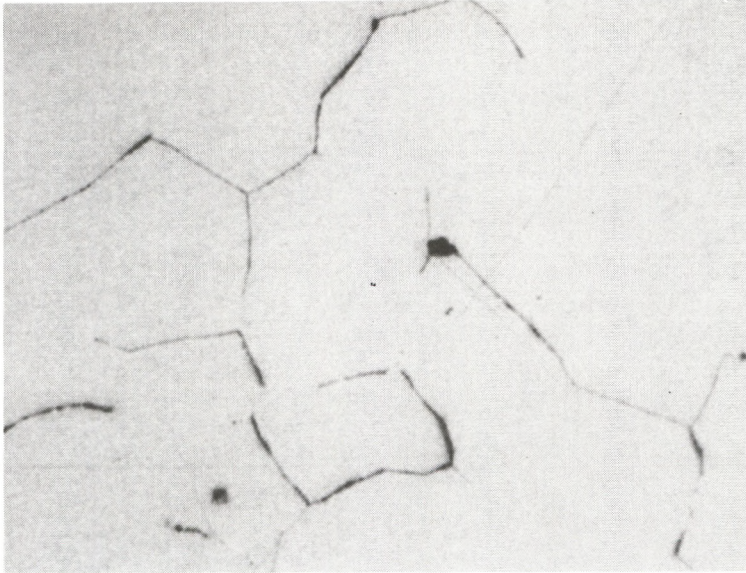


Figure 4. Grain boundary impurity in 18ct MBG. Mag x 800.

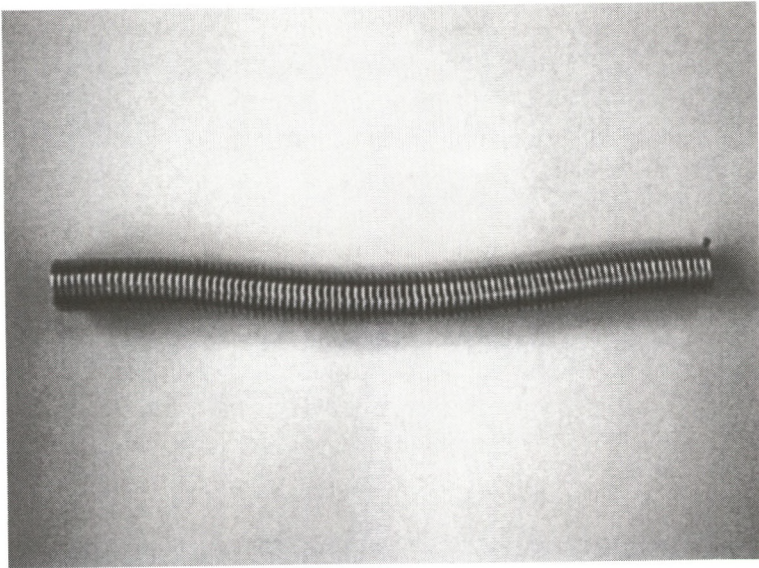


Figure 5. Hoop earring coil.

much higher. Microscopic particles of lead picked up on the surface of the workpiece were sufficient to result in subsequent processing difficulties. Post contamination heat treatment operations - soldering, annealing or hardening for example - have resulted in lead diffusion via the grain boundaries. Prior surface cracking was not necessary for this to occur, alloying readily taking place resulting in a rapid decrease in strength and ductility in the affected areas. Further examination of the workpiece under a high magnification stereo microscope confirmed the presence of lead debris at the surface and diffusion into the surface.

The customer was immediately made aware of our findings and the failure mechanism explained with the aid of micrographs and the relevant analysis traces. The customer confirmed that this and other jobs had been manufactured using lead formers during the raising operation. Some craftsmen used tissue paper as an interleave between the former and the workpiece however this tore easily and was not always used. All workpieces were cleaned prior to any heat treatment operation in order to remove any lead debris remaining after the forming process. Any damage to the formers will potentially deposit debris on the workpiece - often not visible to the naked eye. This residual debris was the origin of the contamination identified. Subsequent annealing has led to diffusion through the grain boundaries resulting in the brittle grain boundary phase. Minimal cold work was required to result in failure. Although the customer insisted the workpiece was cleaned prior to annealing this was not to an acceptable level. It was suggested that a more resilient interleave was used to minimise any surface contamination since the customer intended to continue using lead formers. It is believed a more resilient grade of paper is used and particular attention paid to cleaning workpieces prior to any heat treatment operation. To date there has not been a reported recurrence of this problem - once the failure mechanism had been identified and explained to the customer appropriate action was taken to prevent this. Without the technical analysis further expensive failures may have occurred.

The next case study also involves surface contamination but of a



different nature. The effects were not as catastrophic however an equally unsaleable product was the end result.

### **Surface contamination of 9ct hoop earrings.**

This customer manufactures hoop earrings from thin strip. They produce a large number of designs in plain, patterned and diamond cut styles and in a variety of carats and colours. All are manufactured from thin strip typically 0.004" thick which has been sheared to high tolerances. The bulk of the product is manufactured from an alloy known as 9ct DF - nominally 37.5% Au, 10.0% Ag, 44.5% Cu, 8.0% Zn - which is the general working 9ct yellow alloy used throughout the UK industry. Strip is supplied to the customer annealed and within the hardness specification 110 - 115HV. The manufacturing process involves pulling the strip through a pre-form die and winding the resultant "tube" onto a helixed cylindrical mandrel. This results in a "coil" of hoop earrings (figure 5). The coils are cut into individual earrings using a diamond bladed saw and passed through a conveyer furnace at 600°C to stress relieve prior to assembly into finished items. Once stress relieving has been completed caps and pins are soldered in place and the completed items subjected to a intensive finishing process. Any diamond cutting necessary is carried out prior to final inspection and despatch. The customer runs a highly professional operation and produces large quantities of earrings predominantly for the UK market.

The problem brought to the attention of Technical Services was surface related. After finishing random "blemishes" were evident on the surface of the earrings (figure 6). Additional finishing of the bulk product was unsuccessful in removing the blemishes resulting in large quantities of scrap product. This did not appear to be particular to any size or style of earring and was, according to the operators, increasing in frequency. The customer was certain this was a problem associated with the strip - possibly rolling defects. Strip held in stock for this customer was inspected; no external defects or inclusions were identified and the strip appeared to conform to all order requirements. Examination of returned



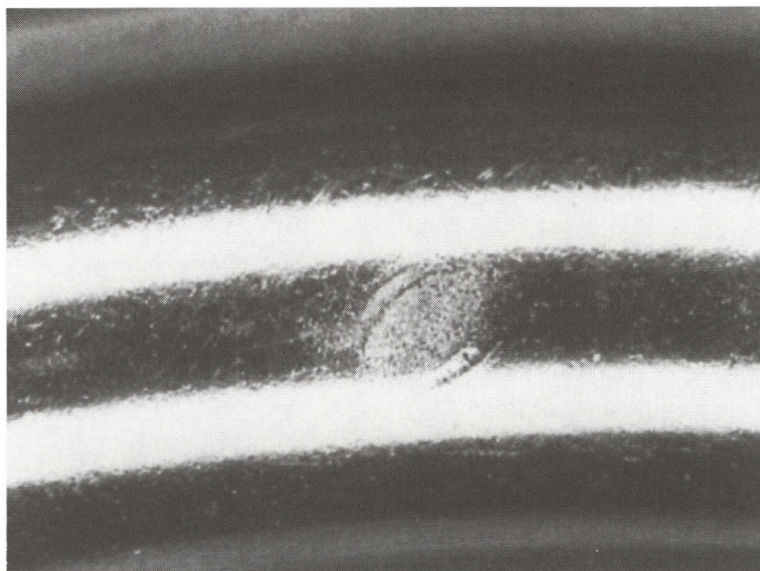


Figure 6. Surface blemish on 9ct hoop earring. Mag x 30.

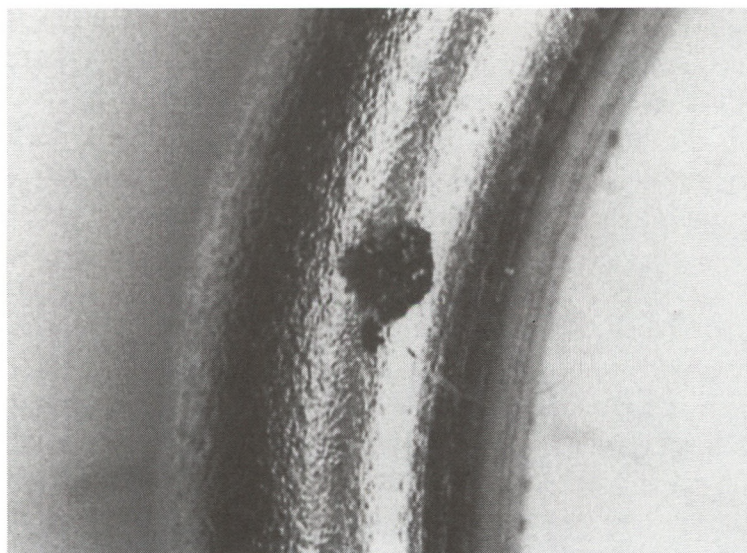


Figure 7. Surface inclusion on 9ct hoop earring. Mag x 20.

earrings revealed a reaction present in the blemish area suggesting that a surface reaction was occurring post delivery.

A member of Technical Services carried out an on-site process audit to determine at which stage the defect originated. Inspection of the delivered strip again confirmed no visible defects or inclusions present. On completion of the forming process several coils were closely inspected with an acceptable surface finish evident. The coils were cut, re-inspected as separate earrings and passed through the conveyer furnace for stress relieving. On inspection several were noted to have black surface inclusions present (figures 7&8) that were difficult but not impossible to see with the naked eye. After finishing the inclusions had been removed resulting in blemishes typical of the problem reported. It appears debris from the interior of the furnace was being deposited on the earrings during the stress relieving process. The customer was immediately advised to clean the furnace interior before any further product was processed. Samples were taken in order to perform further analysis on the inclusions and identify their origin.

Analysis at Technical Services by X-ray fluorescence gave inconclusive results. Microprobe analysis by scanning electron microscope revealed the inclusions to be rich in zinc, iron, aluminium, silicon and carbon; potassium and chlorine were also detected. Examination at higher magnification using the electron microscope revealed the presence of microscopic swarf (figure 8). This was indicative of general furnace debris that will deposit on the inside of the tube over time and has originated from several areas:

- Zinc has volatilised from previously stress relieved earrings and has condensed on the inside of the furnace tube.
- Iron has originated from the furnace tube. Further examination of discrete surface inclusions revealed the presence of nickel and chrome confirming a nichrome type steel - a family of alloys used for furnace tubes (figure 9).



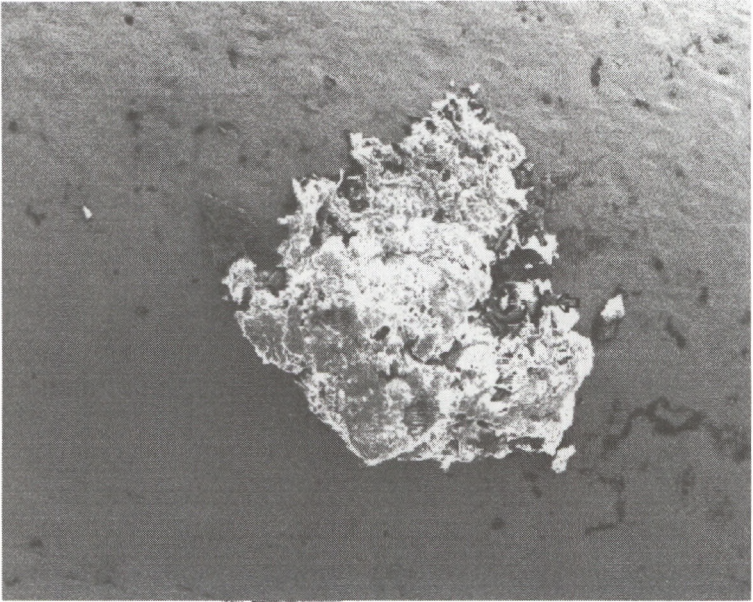


Figure 8. Surface inclusion showing swarf. Mag x 95.

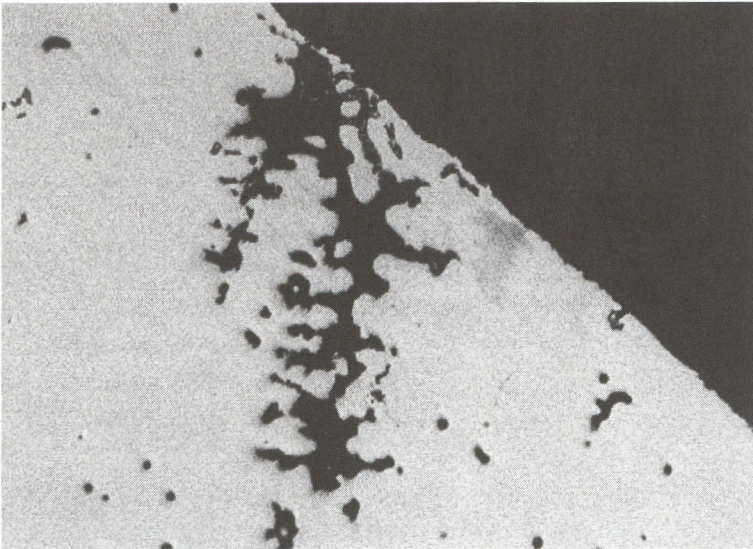


Figure 10. Shrinkage porosity in a 9ct red casting. Mag x 320.



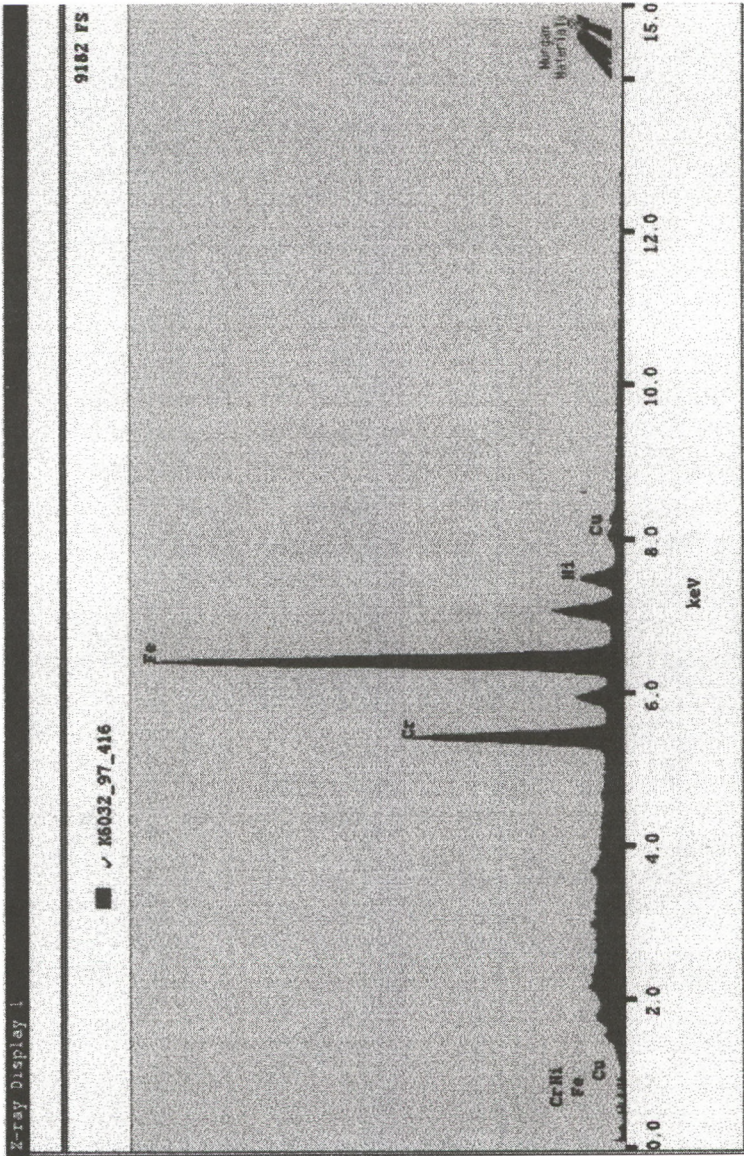


Fig. 9

- Aluminium and silicon were present as a result of debris from the furnace liner bricks.
- Carbon has originated from several sources, the most common being burnt off oil deposits on the earrings remaining from the coiling process.

When deposits reach a certain level they become detached from the furnace tube and fall onto the earrings. Furnaces that are used to heat treat zinc containing alloys require regular cleaning - usually by brushing - in order to remove any deposits that have built up on the inside of the furnace. A further precaution is to ensure the furnace is purged by passing a copper bar through - ideally each morning before any product is processed. This will scavenge any zinc in the furnace - zinc will readily react with the copper block. This block needs to be scoured prior to use to ensure "virgin" copper is presented to the furnace interior. It is difficult to prevent the zinc volatilisation in practice; zinc has a high vapour pressure and as such furnace conditions required will be difficult to obtain. Considering there is a moving belt and workpieces in the furnace together with a moving gas atmosphere and convection effects there is little practical chance of preventing zinc deposits on the furnace tube interior. It has been suggested that possibly a protective coating - such as Argotect (5) - may be utilised to prevent surface exposure and therefore any volatilisation however since this will require removal after the operation it is not a practical option when the quantities of earrings being produced is considered. Regular cleaning of the inside of the furnace by brushing will be sufficient to prevent further build up of other debris.

These findings were explained to the customer and possible solutions discussed. It was agreed that the most practical option was to purge the furnace every morning prior to processing with a scoured copper block and to regularly - ideally once a week - brush the furnace interior to remove any build up of debris. Again, since the suggested actions were implemented there has been no reoccurrence of this



problem.

### **Poor colour reproduction in a 9ct red casting alloy.**

In this case a customer requested technical support after encountering problems casting outwork for a London jewellery producer. The outworker had been supplied with a standard red casting alloy 9ct AA460 - nominal composition 37.5% Au, 5.0% Ag, 57.5% Cu - and was contracted to produce fine section rings. The alloy has a melting range of 900-970°C; the rings were cast using 100°C superheat and a mould temperature of 650°C. A centrifugal casting machine with optical pyrometry was used and the melt covered by 90/10 nitrogen/hydrogen. After de-investment it was noted that the cast trees were very black. Acid pickling and cleaning resulted in pale yellow and completely unacceptable castings. Technical Services was asked to determine why there was poor colour reproduction - it was suggested that the grain supplied was not up to standard and the customer was requesting replacement with “conforming” product. After assurance that immediate analysis would be carried out on the sub-standard castings the customer was advised to withhold further casts using this alloy until the results of our investigations were known.

Chemical analysis of stock grain from the same batch confirmed the composition was within standard tolerances with no deleterious impurities detected. Examination of the submitted castings confirmed poor colour reproduction to the original red alloy. The tree showed indications of residual copper oxide and copper rich surface areas. Random castings were microsectioned for metallographic analysis. The following defects were identified:

- Excessive shrinkage porosity throughout the casting section (figure 10). Examination of the spruing system suggested this was adequate for progressive solidification to occur.
- Excessive levels of copper oxide present throughout the section

(figure 11).

- The presence of several large spherical gas pores (figure 12). Closer examination at higher magnifications revealed the presence of gas microporosity throughout the section.
- Analysis at higher magnification revealed the presence of a copper depleted layer at the surface. Etching the microsections with alcoholic ferric chloride confirmed this (figure 13).
- Analysis of the casting surface by X-ray fluorescence confirmed a reduced copper content.
- The presence of a chlorine reaction at the surface of the castings (figure 14). This was identified by microprobe analysis on the electron microscope.

The above defects are indicative of overheating the melt prior to casting. There was a heavy oxide layer generated on the surface of the castings as a result of the high copper content of the alloy and a high casting temperature. Prolonged acid pickling was required to remove the layer resulting in a copper depleted surface, the pale yellow colour being characteristic the gold, silver and zinc rich surface remaining. When subjected to a vigorous polishing operation the original colour returned.

The customer was informed of the findings and the reasons discussed. It was accepted that the grain conformed to order requirements and the casting process required optimisation for this alloy. The customer agreed to change his casting parameters in order to prevent the formation of a heavy oxide layer. There was the suggestion that the calibration of the optical pyrometer be checked since the stated 100°C superheat would not normally result in the defects identified. The presence of the chlorine reaction is unusual. It appears to be coincidental with surface porosity suggesting chlorine has been introduced in liquid form and entrapped. The customer insisted that all casting trees are pickled in hydrofluoric



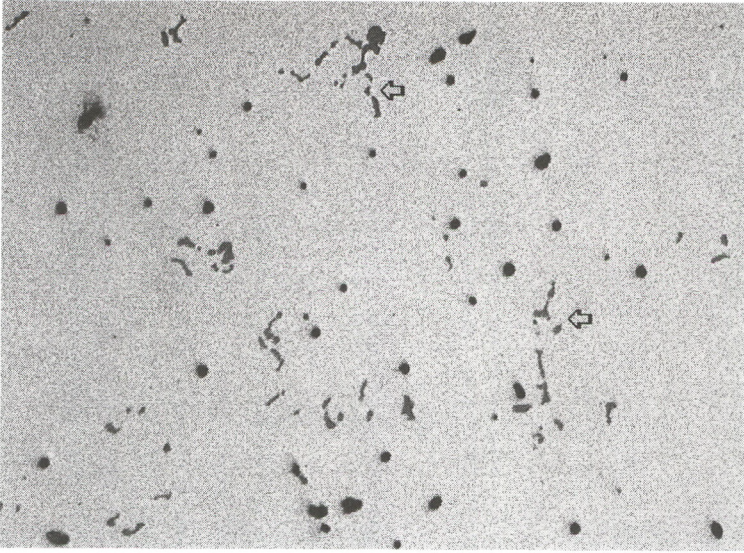


Figure 11. Copper oxide in a 9ct red casting. Mag x 320.

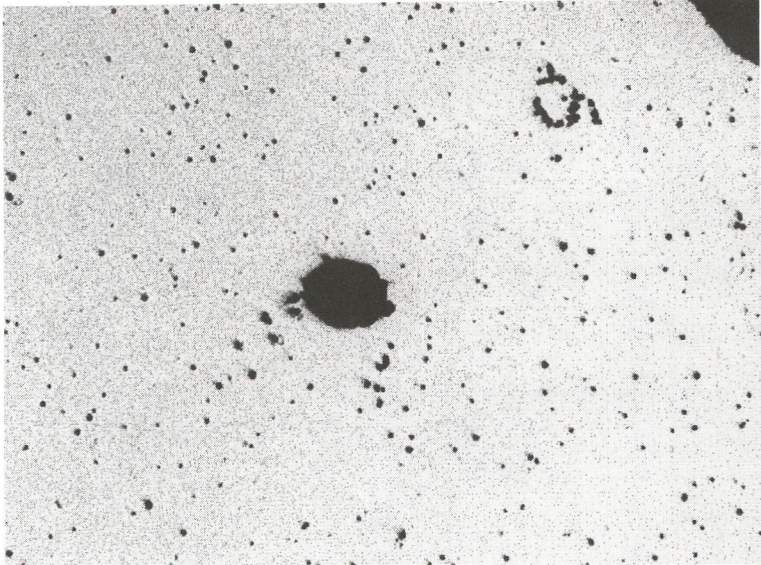


Figure 12. Gas porosity in a 9ct red casting. Mag x 160.

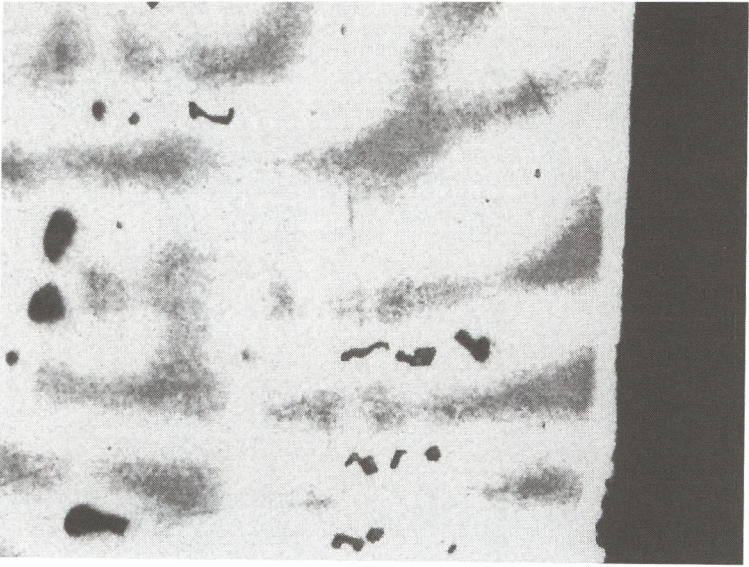


Figure 13. Highlighted surface layer in 9ct a red casting. Mag x 1280.

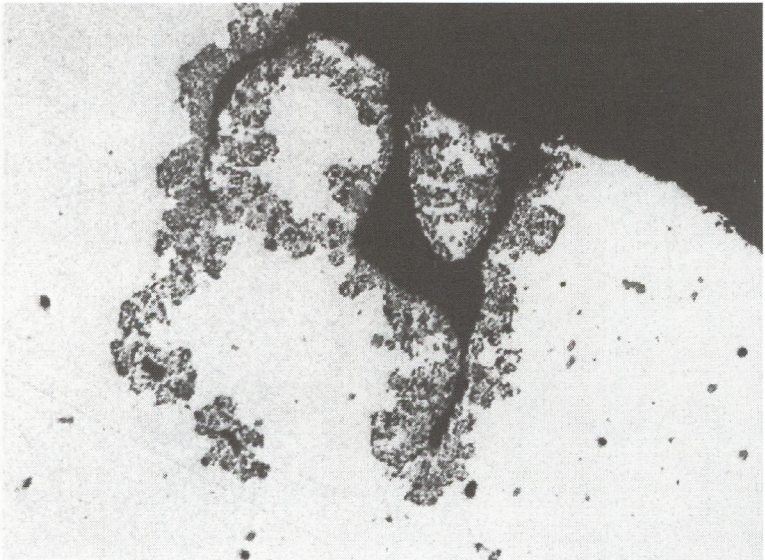


Figure 14. Chlorine reaction in a 9ct red casting. Mag x 320.



acid and at no time come into contact with hydrochloric acid. The reaction was identified prior to etching the microsection in alcoholic ferric chloride in the laboratory negating any possibility of cross contamination from this source. It can only be assumed that the casting has been immersed in a chlorine containing liquid at some stage during processing. This is not immediately detrimental however will result in corrosion products discolouring the surface of the castings. There is also the possibility that these relatively high concentrations of chlorine may lead to stress corrosion failure. New grain was supplied and after minimal trialing successful castings were produced.

The following case study demonstrates problems may be easily resolved, quick analysis saving production time and money.

### **Poor soldering of 14ct solder filled chain links.**

This customer manufactures bracelets and necklets. Part of their product range involves the production of bracelets from individual links that are assembled into finished products using pins. The links may be twisted after soldering to produce different designs therefore a minimum strength is required. The links are manufactured from solder filled wire in the form of tape. This will be familiar to chain makers however for the benefit of others a brief overview on the production and use of solder filled wire follows.

Solder filled wire is a case alloy tube with a solder alloy core. Primarily it is used for chain making. The majority of chain produced today is manufactured from solid wire and subjected to a solder powder application operation - usually by immersion in a slurry (6). Excess solder powder is removed from the chain surface and the chain soldered, typically by passing in hank or single strand through a conveyer furnace set between the liquidus of the solder and the solidus of the case material. Some chain styles may be difficult to join using this method - the design may be of a more complex nature or the wire gauge particularly fine - necessitating the use of solder filled wire. After making chain solder

application is not required therefore it may be tinned - to prevent sticking - and passed directly through the soldering furnace. Once liquid the solder flows outwards from the core; due to the capillary action induced by the joint geometry it fills the joint area creating a strong link on solidification. This process is generally accepted as being the more expensive of the two. Solder filled wire may also be used for jump rings and posts amongst other applications. Production of solder filled wire usually follows one of two routes:

- A blind hole is machined longitudinally down the centre of a solid billet of case alloy. A solder rod is inserted into this hole and the complete assembly fired to bond the solder to the case. This may then be rolled and drawn to the required size and shape.
- A rod of solder alloy is inserted through the centre of a tube of case alloy. This is then rolled and drawn to size, the process ensuring a cold pressure bond is created between the case and core. This bond is important - at smaller gauges any lack of case/core bond may lead to failures.

Both the above methods have advantages and disadvantages, the type used being down to the manufacturers preference. The solder filled wire under discussion was produced via the latter method.

14ct solder filled wire had been supplied to produce individual links for bracelet assembly. The problem experienced was poor soldering performance on the most recent batches of tape delivered. Links appeared to be formed to a satisfactory standard but after passing through the soldering furnace a high percentage had solder "balling" at the joint (figures 15&16). Links that appeared to have soldered satisfactorily had below standard joint strength. The customer returned links and as-supplied wire for examination requesting the assistance of Technical Services - it was their belief that the wire was at fault but welcomed any advice if this proved not to be the case.



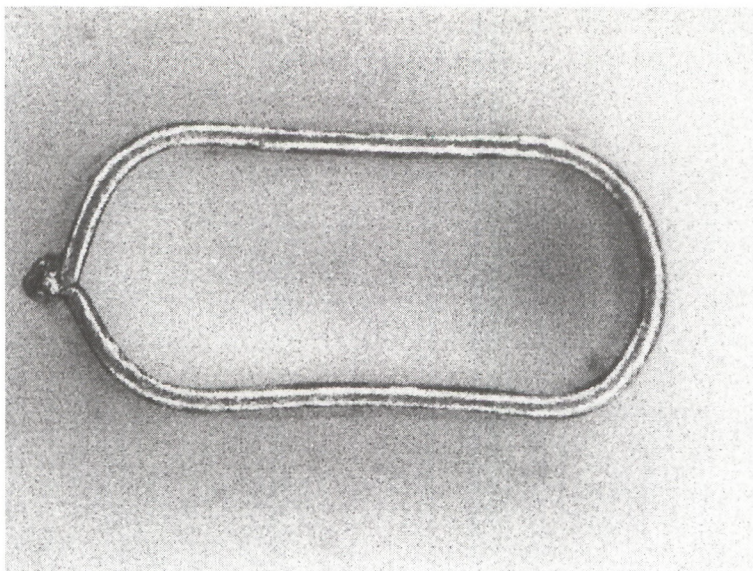


Figure 15. Solder “balling” on a 14ct link - plan view. Mag x 30.

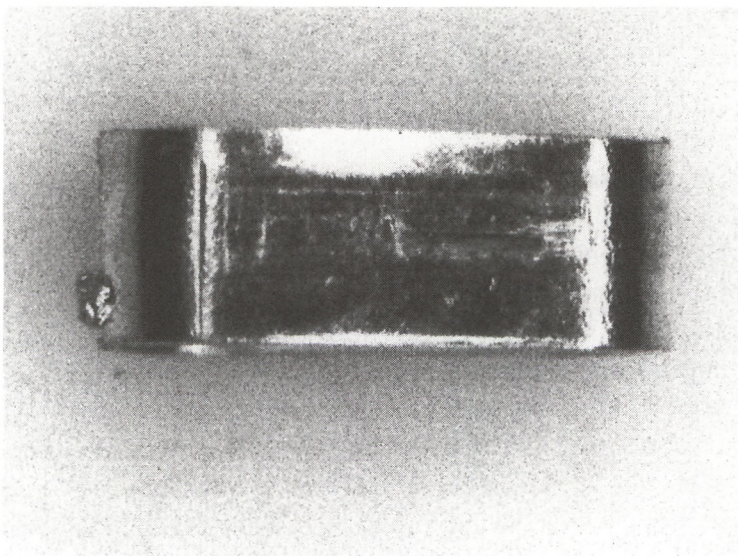


Figure 16. Solder “balling” on a 14ct link - side view. Mag x 28.

As-supplied wire was tested. Tensile properties, grain size and hardness all conformed to order requirements.

	UTS (N/mm <sup>2</sup> )	Elongation (%)	Grain Size (mm)	Case Hardness (HV)
Nominal	440 ± 15	35 minimum	0.025 maximum	100 ± 5
Measured	450	38	0.023	103

The solder core was inspected in cross section and although not central was of the correct proportion and shape. The rejected soldered links were examined under the stereo microscope; links that had no “ball” present appeared to have an excessively large solder fillet - this was confirmed in microsection (figure 17). Examination of the submitted unsoldered links revealed the following:

- Unsuitable joint geometry (figure 18). The ideal joint geometry for links manufactured from solder filled wire is parallel faces. If equipment, wire or design limitations will not allow this then any resultant “V” must be open towards the inside of the link. In this case the joint faces were neither parallel nor the resultant “V” facing the inside of the link. The geometry had discouraged even solder flow through the joint and lead to the formation of a large solder fillet. The resultant link was weakened due to the relatively brittle nature of this excess solder present in the joint area. It was unlikely this geometry was the result of the wire properties - there was no evidence of springback occurring. It was probable that the chain machine required re-setting.
- The joint faces were smeared (figure 18). This resulted in poor solder flow - the solder core exits clearly evident on the link faces and not on the joint faces. Balling of the solder core had resulted with poor flow into the joint area. This was either the result of the wire being too soft or the cutter being blunt. Testing had confirmed the tape conformed to hardness requirements therefore





Figure 17. Excess solder fillet in a 14ct link. Mag x 320.

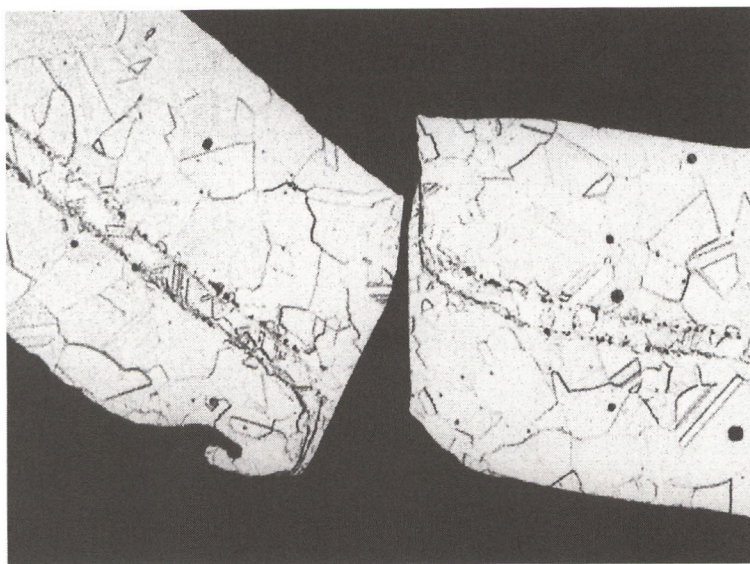


Figure 18. Poor joint geometry and smearing in a 14ct link. Mag x 320.

the cutter required inspection and re-sharpening if necessary.

The customer was immediately informed of our findings. Inspection of the machine confirmed the cutter was blunt. This was sharpened to produce a satisfactory cut; further fine tuning produced satisfactory joint geometry. An inspection and maintenance routine was put in place to prevent reoccurrence. The customer agreed although they would eventually have arrived at the same conclusions, it would have taken far longer and wasted more expensive production time and scrapped product. The involvement of Technical Services was key to a quick and acceptable outcome.

All case studies discussed so far have dealt with carat gold alloys. The following study remains within the surface related theme but involves standard silver.

### **Enamelling of a standard silver badge of office.**

A badge of office manufactured from 1.8mm thick standard silver sheet was returned stating the sheet supplied was sub-standard. The badge consisted of an oval stamped from sheet with a design cut and enamelled (figure 19 ). The customer stated there were areas of "contamination" present causing discolouration and flaking of the enamel. Particular emphasis was placed on an area of contamination present on adjacent faces of the workpiece suggesting to the customer that it was through-section. Sales requested analysis to determine whether the contamination originated from the supplied sheet or the customers' process.

A brief overview of the enamelling process follows for those who are unfamiliar with it:

- The workpiece to be enamelled undergoes any soldering operations required and is subjected to a final anneal to stress relieve.



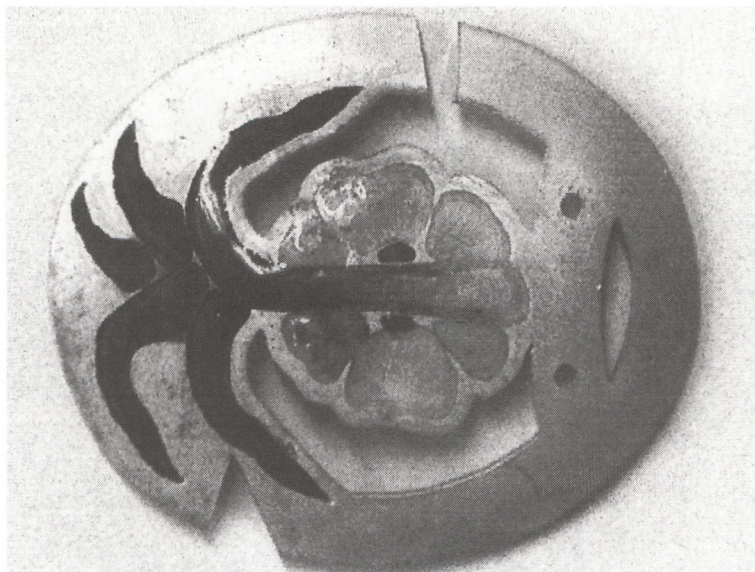


Figure 19. Standard silver badge of office. Mag x 1.2.

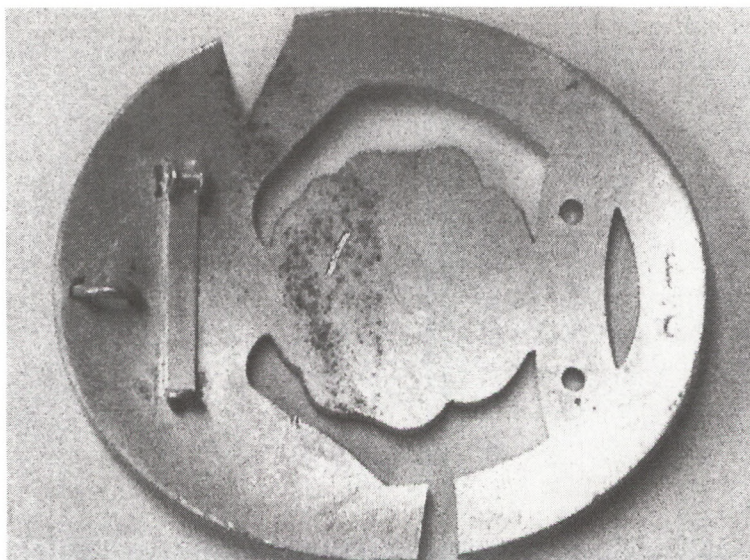


Figure 20. Discoloured rear surface. Mag x 1.2.

- The workpiece is cleaned, usually by one of two methods: (i) acid pickle in nitric acid to remove oxides, dirt and grease or (ii) electro stripping (7)(8). Some enamellers prefer to use sulphuric acid - it is less reactive - however nitric acid should result in a better surface. Neutralising in an alkali bath completes this stage of the process.
- The enamel is applied as a paste and is dried.
- The workpiece is fired at 800°C. Care must be taken since the solidus of standard silver is 805°C.
- Further coats of enamel are applied if required, treating the workpiece between coats.

Initial examination of the workpiece confirmed the presence of a black “contamination” on top and bottom surfaces resulting in metal discolouration (figures 19&20). Areas of enamelling were discoloured and flaking with poor adhesion. Microsections of the contaminated and non-contaminated areas revealed the following:

- Partial liquation of the workpiece. Through section porosity typical of incipient fusion was present (figure 21).
- The presence of oxide extending into the workpiece via surface porosity suggesting liquation had occurred prior to or during the oxidation process.
- Areas not discoloured had little or no surface porosity (figure 22).

The workpiece had been heated above the solidus of standard silver resulting in grain boundary liquation. Pickling acids used in the enamelling process had become entrapped in surface porosity and resulted in corrosion products discolouring the surface. These will be hastened - and blackened - by the subsequent heating operation when



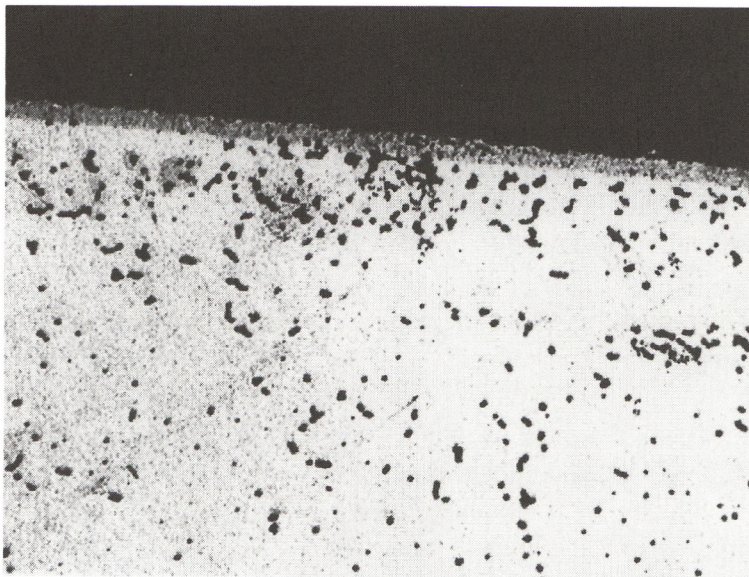


Figure 21. Surface porosity in “discoloured” area. Mag x 80.

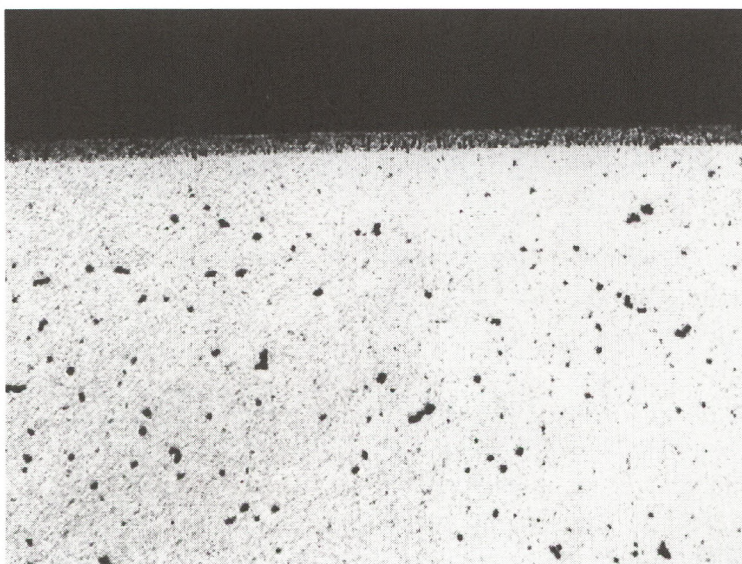


Figure 22. Surface free of porosity in “clean” area. Mag x 80.

enamelling. Further analysis by scanning electron microscope was possible however was not determined necessary. The discolouration originated from acid entrapment in surface porosity, confirmation of the pickling acid used being irrelevant. The customer was unavailable for comment in this case. A copy of the technical report was forwarded however no further correspondence was received. It was assumed that our advice was taken and used to good effect.

The report suggested an overview of the techniques used be made in order to prevent recurrence:

- Any heat treatment must be performed in a shaded area if a torch is used. Metal colour can then be accurately judged and the possibility of overheating minimised. It is not uncommon to overheat alloys - particularly silver - on sunny days if adequate shading is not used.
- Workpieces should be pickled in nitric acid prior to enamelling. All acid residues must be removed after this process - the use of an alkali neutralising solution is recommended and preferably the use of an ultrasonic bath. All entrapped acids will eventually result in corrosion products.

The final case study is an important lesson in investigating internal techniques before requesting further help. The example demonstrates that sometimes with a little forethought and insight we can help ourselves.

### **“Failure” of 9ct ½ hard tape at goods inwards inspection.**

The customer takes delivery of 9ct DF wire in tape form for manufacture into links then bracelets and necklets. Annealed wire is unsuitable for use in the particular machine used since it has a tendency to concertina in the feed blocks and stop production. Wire is supplied in the ½ hard condition - 20% cold work - and must meet the order



specification:

Hardness : 180 → 200HV

Elongation after fracture : 20 → 25%

Our manufacturing process to convert round wire into rectangular tape requires the feedstock to be in the hard condition. The ½ hard temper is achieved by let down anneal and can present production problems. The formation of a cast in the wire when reeling will result in “pig-tailed” wire which again presents feeding problems during link manufacture. This can be overcome however it is a slow process compared to standard final annealing meaning any rejected material is very costly.

Wire is usually supplied in the annealed condition for link manufacture. Normally in the ½ hard condition it would be unsuitable due to “springback” - the closed ends of a link spring apart after formation making subsequent joining of the link impossible. Springback does not present a problem in this case - links are joined during forming by argon arc welding before being released from the jaws.

The customer has a goods inwards inspection programme to ensure all supplied product conforms to ordered requirements. None conforming product is returned to the supplier for replacement. The particular problem under discussion was brought to the attention of Technical Services after several deliveries of this wire uncharacteristically failed inspection and was returned for replacement. The customer was now becoming desperate to complete orders and needed conforming product to do so. The customer determined the supplied wire had elongations of 2-3%. Returned reels were matched to our internal final inspection records which stated elongations ranging from 22 - 25% measured. The wire was re-tested and passed conformance. Technical Services requested a site visit in order to inspect the test facilities. Several “failed” reels were re-tested on the customers tensometer and 2% elongation measured. At this point comments were made that our supplied annealed wires were also showing lower

elongations than expected. After several tests it became apparent that all samples were failing where the wire exited the top jaws of the tensometer. The top and bottom jaws were switched and the next samples tested all failed where the wire exited the same - now bottom - jaws. It was concluded that the jaws were damaged resulting in false elongation values. Any damage caused to the wire will act as a stress raiser leading to premature failure. This will have a greater effect in the case of  $\frac{1}{2}$  hard compared to annealed wire due to the critical crack length - at which catastrophic failure occurs - being smaller for this condition. Wire was trialed and produced acceptable links. The customer examined the tensometer jaws, made some slight adjustments, and further tests resulted in acceptable elongation values.

This study demonstrates when problems are encountered, investigate logically before requesting outside assistance. Is there anything that appears out of the ordinary? Will the supplied product run on the machine even though it failed inspection? If so is inspection at fault? Was the material ordered correctly? Technical Services was only too pleased to assist in this case however forethought may have negated the need for the site visit.

### **Conclusion.**

The aim of this paper was to demonstrate that involving the Technical Services Department of your materials supplier can save time and money. Production problems and rejections are rarely straightforward but always costly to the manufacturer. These must be kept to a minimum and only jewellery that is fit for purpose sold to retailers and the public. It is essential that a partnership exists between manufacturers and suppliers to ensure that the most suitable alloys are supplied in the required conditions for a given process to succeed and product to be economically produced. This partnership must naturally extend into problem solving when necessary. The industry can only benefit from exposing and sharing these problems, this symposium being one forum in which this can occur. None of the case studies discussed are intended



to belittle the manufacturers concerned. For professional reasons none have been named however they are thanked in advance for allowing the use of their short-term misfortunes to help educate others.

In my experience manufacturers' problems are often due to their own processes and conditions rather than any inherent fault in metal supplied. There is still a widespread technical ignorance of the materials and processes used within the industry today. Suppliers can play an important role in providing the after-sales services discussed in this paper and in doing so help to educate manufacturers to a higher technical level. Problems encountered should be used as an opportunity to learn more about the materials and processes being used. Exposure to the analytical techniques discussed in this paper is very important in order for manufacturers to understand what can be achieved. Without the facilities of an analytical laboratory problems may not be identified and categorised correctly resulting in the repetition of costly mistakes. If a supplier does not provide technical back-up but replaces metal - even free of charge - this can only be seen as a short term advantage and can be very expensive, resolution to problems generally arising through trial and error. Manufacturers must insist on full technical back-up from their supplier and suppliers must be prepared to honour this - your customer will soon realise they are receiving full value for money and that can only result in increased sales.

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