

# “But I’ve always done it this way.” Technical support – it makes a difference\*

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## Summary

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 and 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 Europe and the USA has over recent years become increasingly technology based. 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.

The aim of this paper is to 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.

A typical Technical Services Department will consist of several 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 should be strictly objective in nature, the aim being to resolve problems to a satisfactory conclusion and not to deflect blame away from the supplying company. It can be considered a free technical consultancy service. Advice will be commonly sought on alloy and process selection, the majority of cases being dealt with over the telephone; however, site visits should 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. This paper will discuss four examples based on the type of problem frequently encountered within the jewellery manufacturing industry by the author. Problem solving by manufacturers is generally based on surface analysis – either by eye, magnifying 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.

\* Footnote: This paper is adapted from one given at the 12th Santa Fe Symposium, Albuquerque, May 1998

- 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. All samples should be 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 must also be 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 the Technical Services department is usually advisable and samples may be taken at this stage.

Certain information is required 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?
- 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. It is noted that, in the majority of cases, the customer is extremely appreciative that someone has taken time to give technical assistance.

Requests received for advice and analysis may be wide ranging; therefore, the examples chosen are all – with one exception – based loosely on surface related problems.

### Surface contamination of 18ct gold hand raised sheet

This first example involves the appearance of cracks in the corners of a workpiece raised from 18ct yellow sheet. It was suggested that the supplied sheet was not 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. The hardness of this alloy is HV 125 in the annealed condition; however, an alloy of similar colour but with a lower annealed hardness and increased ductility exists – 18ct MBG. This has a slightly higher gold content (closer to 19ct) and a small zinc addition but retains the preferred 2N colour. However, when considering the intended market for high quality, hand raised workpieces, the slight increase in caratage should not have an impact on profitability. There are other 18ct alloys available with increased ductility to 18ct HB and MBG but 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 alloy has a lower work hardening rate.

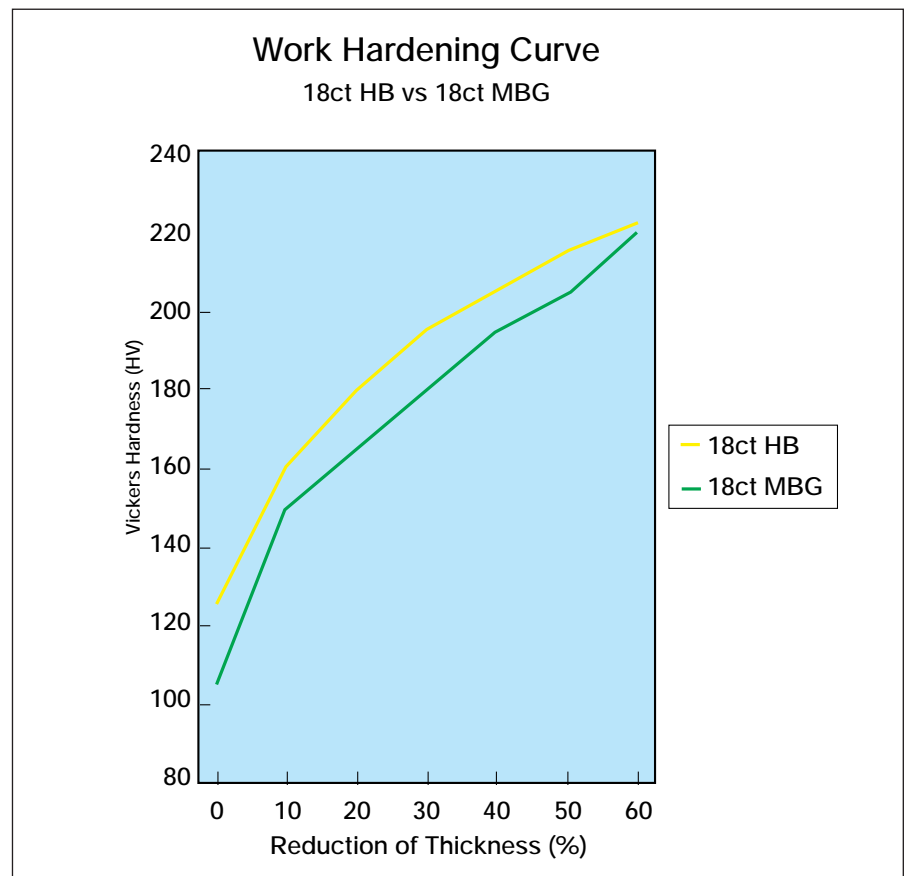


Figure 1 - Work hardening curves of 18ct HB and MBG alloys



**Figure 2** - Intergranular cracking in 18ct yellow gold alloy HB sheet. x30



**Figure 3** - Presence of grey phase at grain boundaries in 18ct HB alloy sheet. x800



**Figure 4** - SEM examination showing lead debris on surface of 18ct HB alloy sheet. x40

Workpieces produced from these alloys may not always be items of jewellery as such but “object d’art” and 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 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.

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 but the 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 HV105 maximum hardness 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; examination at higher magnifications revealed the presence of a grey grain boundary phase, Figure 3. Analysis by X-ray fluorescence (XRF) and atomic absorption spectroscopy (AAS) 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 but 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 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, Figure 4.

These findings were discussed with the customer and the failure mechanism explained with the aid of micrographs and the relevant analysis traces. It was confirmed that this and other jobs had been manufactured using lead formers during the raising operation. Tissue paper is often used as an interleave between the former and the workpiece but this may tear easily. 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 it may have been perceived that the workpiece was cleaned prior to annealing, this was not to an acceptable level. It was suggested that a more resilient interleave be used to minimise any surface contamination 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 has been identified and explained to a customer, appropriate action may be taken to prevent reoccurrence.

The next example also involves surface contamination but of a different nature. The effects were not as catastrophic but an equally unsaleable product was the end result.

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

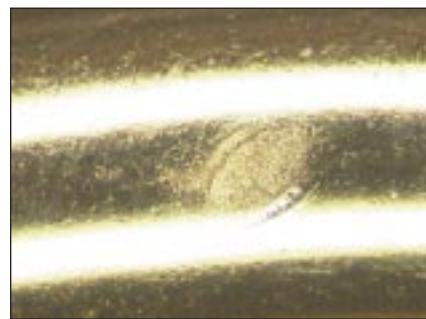
This example highlights a problem encountered with 9ct hoop earrings manufactured from strip. The product is manufactured from an alloy known as 9ct DF, nominally 37.5% Au – 10.0% Ag -44.5% Cu -8.0% Zn, which is the general working 9ct yellow alloy used throughout the UK industry. Strip is supplied to the customer in the annealed condition (specification hardness HV110-115) and is pulled through a pre-form die, the resultant “tube” being wound onto a helixed cylindrical mandrel.

This results in a “coil” of hoop earrings. The coils are cut into individual earrings using a diamond bladed saw and passed through a conveyer (belt) furnace 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.

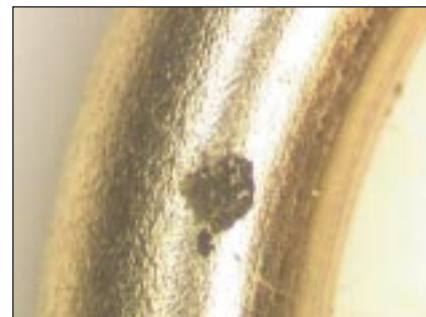
After finishing, random “blemishes” were evident on the surface of the earrings, Figure 5. Additional finishing of the bulk product was unsuccessful in removing the blemishes, resulting in large quantities of scrap. This did not appear to be particular to any size or style of earring and was increasing in frequency. Strip held in stock for this customer was inspected; no external defects, such as roll marks or inclusions, were identified and the strip appeared to conform to all order requirements. Examination of returned earrings revealed a reaction present in the blemish area suggesting that a surface reaction was occurring post delivery.

An on-site process audit was carried out 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. Inspection at this stage revealed several had black surface inclusions present that were difficult but not impossible to see with the naked eye, Figure 6. 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 immediate advice given was 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.

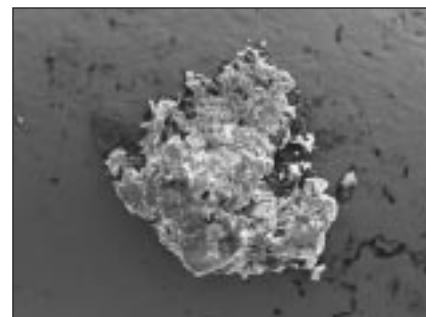
Analysis at Technical Services by



**Figure 5** - Blemish on the surface of a 9ct DF alloy hoop earring. x30



**Figure 6** - Inclusion on surface of 9ct DF alloy earring after annealing in belt furnace. x20



**Figure 7** - SEM examination showing swarf on surface, 9ct DF alloy. x95

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 7. 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.
- 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 a clean “virgin” copper surface is presented to the furnace interior. It is difficult to prevent the zinc volatilisation in practice; zinc has a high vapour pressure and the required furnace condition will be difficult to maintain. Considering that 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 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 brush the furnace interior, ideally once a week, to remove any build up of debris. Since the suggested actions were implemented, there has been no reoccurrence of this problem.

### Poor colour reproduction in a 9ct red gold casting alloy

In this case, problems were encountered during investment casting of a 9ct red gold. A standard 9ct red casting alloy AA460, nominal composition 37.5% Au -5.0% Ag -57.5% Cu, had been supplied for production of 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 fitted with optical pyrometry was used and the melt covered by 90% nitrogen/10% hydrogen gas mixture. 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. The origins of the poor colour reproduction required determination in order to resolve this problem and the customer was advised to withhold further casts using this alloy until these had been identified.

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 of the original red alloy. The cast 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 8. Examination of the gating system suggested this was adequate for progressive solidification to occur.
- Excessive levels of copper oxide present throughout the section, Figure 9.

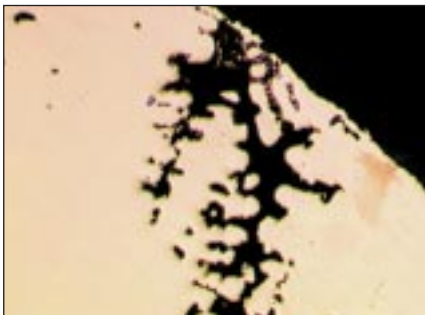


Figure 8 - Shrinkage porosity in cast 9ct red gold alloy AA460. x320

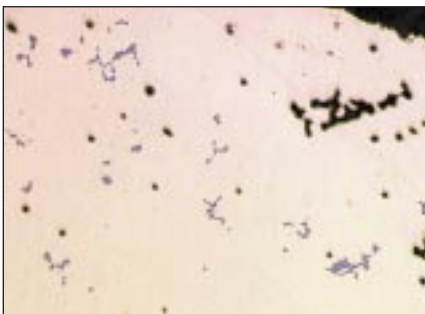


Figure 9 - Copper oxide in cast 9ct gold alloy AA460. x320

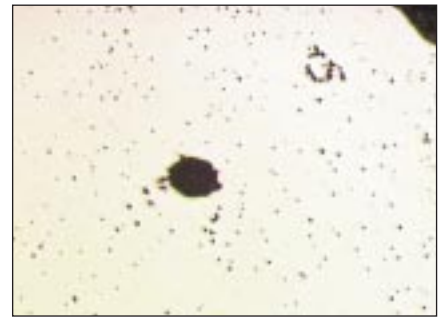
- The presence of several large spherical gas pores, Figure 10. Closer examination at higher magnification revealed the presence of gas microporosity throughout the section.
- Analysis at higher magnification revealed the presence of a copper depleted layer at the surface, confirmed by etching the microsections with alcoholic ferric chloride, Figure 11.
- 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 12, 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 of the gold-, silver- and zinc-rich surface remaining. When subjected to a vigorous polishing operation, the original red colour returned.

The customer was informed of the findings and the reasons discussed. It was accepted that the casting process required optimisation for this alloy, particularly a change in the casting parameters used in order to prevent the formation of a heavy oxide layer. It was suggested 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 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 but 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 subsequently produced.

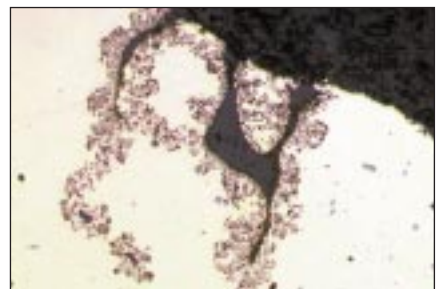
The final case study is an important lesson in investigating internal techniques before requesting further help. The example demonstrates that sometimes, with a little forethought and insight, we can help ourselves.



**Figure 10** - Large gas pores and gas porosity in cast 9ct gold alloy AA460. x160



**Figure 11** - Copper-depleted layer at surface on section etched in alc. ferric chloride. x1280



**Figure 12** - Chlorine reaction at surface of casting, 9ct AA460 alloy. x320

### **“Failure” of 9ct wire at goods inwards inspection**

In this example, 9ct DF wire is supplied with 20% cold work for link manufacture meeting the specification:

Hardness : HV 180 - HV 200

Elongation after fracture : 20 - 25%

The customer has a goods inwards inspection programme to ensure all supplied product conforms to ordered requirements. Non-conforming product is returned to the supplier for replacement. The particular problem under discussion was noted after several deliveries of this wire uncharacteristically failed inspection and was returned for replacement. The customer determined that the supplied wire had elongations of only 2-3%. Returned reels were matched to final inspection records which stated elongations were measured in the range 22-25%. The wire was re-tested and passed conformance. A site visit was requested 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 other 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 at the 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 cold worked wire compared to annealed wire, due to the critical crack length at which catastrophic failure occurs being decreased for this condition. Wire was trialed and produced acceptable links. The tensometer jaws were examined, some slight adjustments made, and further tests resulted in acceptable elongation values.

This example demonstrates that when problems are encountered, logical investigation should be

carried out before outside assistance is requested. 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? Double-checking this information may negate the need for a site visit by the supplier.

### **Conclusion**

The aim of this paper has been 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 where necessary; industry can only benefit from exposing and sharing any problems experienced.

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 simply replaces ‘faulty’ metal, even free of charge, this can

only be seen as a short term advantage and can be a very expensive resolution to problems generally arising through trial and error. Manufacturers should 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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