

# The Effect of Silicon Content vs Quench Temperature on Low Carat Casting Alloys

**STEWART GRICE, Metallurgical Manager,  
Cookson Precious Metals Ltd, Birmingham, England**

## INTRODUCTION

There are numerous carat gold alloys available to the jewellery investment caster ranging from additive-free alloys to those containing small additions of certain elements which aid the casting process. This study is concerned with the latter, in particular low-carat gold casting alloys. 9ct alloys are utilised for the majority of the U.K. jewellery industry and their metallurgy is similar to the equivalent 8ct alloys used in Europe and 10ct alloys used in North America. One element commonly added to aid casting performance is silicon, which not only provides bright, oxide-free castings but also increases flow characteristics. An overview of casting alloys revealed that of those found to contain silicon, additions varied significantly from 0.005% to 0.7% depending on carat and intended use. An overview of the levels of silicon added to 9ct casting alloys in the U.K. revealed additions ranging from 0.05% to 0.7%. These alloys are all relevant in today's market and have their specific uses, however their performance will vary significantly.

Alloys that may be classed as "high-silicon" – for the purposes of this study "high" being  $>0.2\%$  – are generally used for specific applications:

1. For use in operations where intensive recycling occurs. As a general rule it is not recommended to use casting charges with a scrap to new metal ratio greater than 50%, however in practice this can and does occur, in some instances charges of 100% scrap being used. The potential for failures then increases significantly. A "high-silicon" alloy will allow a more aggressive scrap recycling programme to be employed before the alloy becomes "silicon-exhausted".
2. For use as master alloys. The

situation may occur – particularly in the smaller workshop – where scrap from other areas of the manufacturing process – e.g. wire, sheet or tube – may be used to make up the casting charge. These alloys – intended for wrought applications – will almost certainly have no silicon present and as such additions of a high-silicon grain will aid the casting process. The silicon content of the master alloy will be diluted by the scrap/grain ratio i.e. if the master alloy contains 0.5% silicon and is mixed 50/50 with other processing scrap the overall silicon content of the charge will be 0.25%.

3. For use in "high abuse" environments. These alloys can be used to good effect with processes where there is no temperature measurement or control, or where there is ineffective melt protection.

The effects of silicon on the performance of casting alloys has been widely documented and is well understood by the majority of the world jewellery manufacturing industry. This study will concentrate on a specific phenomenon encountered when using this element. As higher silicon levels are approached, detrimental as well as the positive effects may occur, including failure both during and after the casting operation. The study will evaluate the effect of quench temperature on a standard 9ct yellow casting alloy with varying silicon additions and will document any best practice determined. It is not concerned with alloy development, the alloys used being commercially available or close to commercially available compositions, but will concentrate on determining the effects encountered

during the casting process. Mechanical and physical properties of castings will be of secondary consideration to failure resistance during the casting process, these having already been documented to some degree as a function of silicon content, if not quench temperature, on standard alloys.

## BACKGROUND

A study of the binary phase diagrams of silicon show that it is insoluble in zinc, undergoes limited solid solubility in gold and silver and forms a series of single and two-phase solid solutions with copper. There are numerous previously published papers outlining the effects of silicon on casting alloys and carat gold alloys in general (1,2). Normandeau and Roerterink (3) outlined the effect of silicon on various casting alloys and stated that caratage was not the limiting factor but the total (gold + silver) content in weight percent of an alloy. This factor was directly related to the silicon content and a critical level of silicon was defined. Exceeding this critical level resulted in embrittlement and failure on subsequent working operations due to a "brittle network of silicon at the grain boundaries". This level had obvious limitations when looking at the higher carats, however for low carat alloys it was possible to tolerate 0.5% silicon without adversely affecting the mechanical properties, with increases in fluidity noted at 0.1%. Hardness and grain size also increased with silicon addition, and an associated colour change noted. This will not be discussed in this study – it was assumed that any decision regarding alloy colour will be taken at the time of grain purchase. Jackson (4) suggested that levels of 0.4% silicon in 9ct alloys gave rise to embrittlement on

soldering due to the formation of a low melting point silver and silicon-rich eutectic phase. Other references note the importance of grain boundary silicon-rich phases leading to embrittlement (5,6,7); however all were concerned with embrittlement in cast pieces subjected to post-casting exterior forces and not during the actual casting process. The latter area is the purpose of this study.

## OBJECTIVES

It had been noted on a number of occasions when using high silicon containing alloys that, for what were described as standard conditions, a caster may achieve full, partial or virtually no success with a cast, the failures becoming evident after devesting. These alloys were used for a number of reasons, the most common being the use of high scrap recycling rates, an increase in which were noted between 9ct alloys containing 0.1% and 0.2% silicon (8). If these alloy systems were considered further, there was not only the possibility for the formation of a brittle grain boundary silicon-rich phase but also the potential for hot tearing if complete solidification during the quenching/devesting process was not achieved. The potential for these phases to remain in the liquid or partially liquid state at the time of quenching was, for the higher level of addition, considerable. It was necessary to determine if phases were grain boundary in nature or precipitated as distinct phases throughout the structure – i.e. a multi phase system as opposed to a single phase system with a grain boundary impurity present.

## Alloy choice

For the purposes of this study the parent alloy chosen was 9ct DF, a general purpose alloy with approximate 3N colour and used throughout the U.K. industry. It has the nominal composition:

37.5% Au, 10.0% Ag, 44.5% Cu,  
8.0% Zn

which places it safely within the ductile zone as described previously. This alloy is used for all types of manufacturing processes and sold as casting grain with various elemental additions present. The alloy has an Ag' value of 18% with the phases  $\alpha^1$ (Au-Ag) +  $\alpha^2$  (Au-Cu) are

Table 1.

Designation	Alloy 1	Alloy 2	Alloy 3	Alloy 4	Alloy 5
Silicon (%)	0.00	0.10	0.25	0.50	1.00

precipitated when slowly cooled to equilibrium at room temperature (9).

When determining the range of silicon additions to be assessed, a study of the most popular casting alloys purchased suggested additions of 0.1%, 0.25% and 0.5% silicon by weight, with control alloys at 0.0% and 1.0%.

The inclusion of the 1.0% silicon alloy may appear unusual however this provided an upper limit, no reference to a jewellery casting alloy containing such a high addition being found.

## Tree design

To correctly assess the casting trials, several standard tree patterns were used to ensure maintenance of identical conditions for all casts. The three standard trees employed in these trials consisted of:

1. A selection of common designs ranging from thin lightweight rings through heavy rings to complex brooch designs.
2. A selection of test pieces to determine mechanical properties, porosity, surface finish, degree of ring sizing tolerated and solderability. These are all standard test pieces employed in numerous other casting studies.
3. A mesh design for measuring fluidity.

## Trials

For all alloys, trees No. 1&2 were cast at 120°C superheat and quenched at temperatures of 600°, 550°, 500°, 450° & 400°C (measured at the button). Analysis was then performed, where necessary as a function of quench temperature, for each alloy. Tree No.3 was cast at 120°C superheat into a flask at 650°C and quenched at 450°C.

## EXPERIMENTAL

### Investment casting procedure

All flasks underwent an identical investing and burnout cycle to a final casting temperature of 650°C. Standard weight charges of 100%

grain were used for each type of tree and heated to 120°C above the liquidus, held for 1 minute and then cast using a Galloni static vacuum assisted machine. Each casting was held under vacuum for 2<sup>1/2</sup> minutes – 3 minutes for alloy 5 – then removed and placed on an insulated plate where temperature measurements were taken. All flasks were quenched in water, cleaned by power-wash and pickled in a sulphamic acid solution for 10 minutes.

### Analysis

The melting range of each alloy was determined from a cast grain sample by differential thermal analysis. Tree temperatures were determined using type K thermocouples, the cast button from a surface contact probe, the middle and bottom castings from probes through pre-formed holes in the investment. Fluidity was assessed by the degree of fill for the mesh pattern and stone setting prongs for each alloy. Surface finish was assessed under a stereo microscope using x10 magnification. Metallography was carried out on microsections prepared by standard techniques using a metallurgical microscope. Further analysis was performed on a scanning electron microscope using both qualitative and semi-quantitative techniques.

Ultimate tensile strength, 0.2% proof stress and elongation after failure were determined from standard dumb-bell type tensile cast samples. All UTS, 0.2% PS, elongation and hardness values were averaged from six samples. Grain size was determined from identical ring castings using a linear intercept method, taking an average of three tests per casting.

## RESULTS and DISCUSSION

### Melting range:

The melting ranges for each alloy were determined (figure 1). Whilst the increase in silicon addition reduced the liquidus considerably, the solidus was influenced to a far greater degree, expanding the melting range of the alloy. It is believed this was due to the formation of low melting point silicon-rich phase(s).

### Temperature measurement

All flasks remained under vacuum soak for  $2\frac{1}{2}$  minutes prior to removal for cooling. Measurements were taken every 30 seconds after  $3\frac{1}{2}$  minutes had expired (1 minute of cooling after removal from the chamber). Analysis of the data generated revealed that for identical cooling times there was a significant difference in button temperature between the alloys – those containing silicon being much higher than alloy 1, even though lower casting temperatures were used. This must

be directly associated with the rate of cooling for each alloy. Further analysis revealed a decrease in the cooling rate for alloys 2, 3, & 4, alloy 1 exhibiting the fastest cooling rate (figures 2 & 3).

The cooling rate of the casting trees will be governed by several factors:

- the rate of heat quantity flowing between the alloy and the investment i.e. the conductivity of the investment,
- the amount of heat energy radiated from the alloy by the button, and
- the specific heat capacity of the alloy.

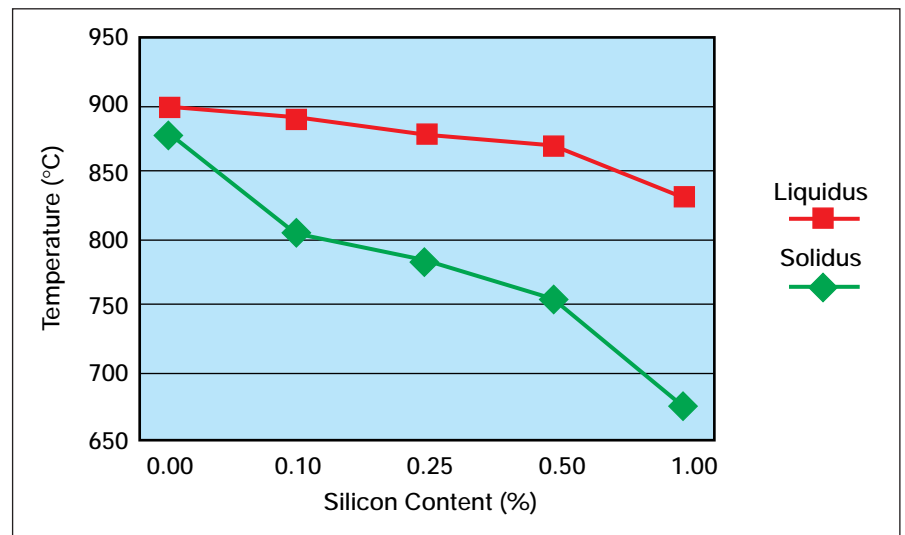


Figure 1 – Melting Range vs Silicon Content

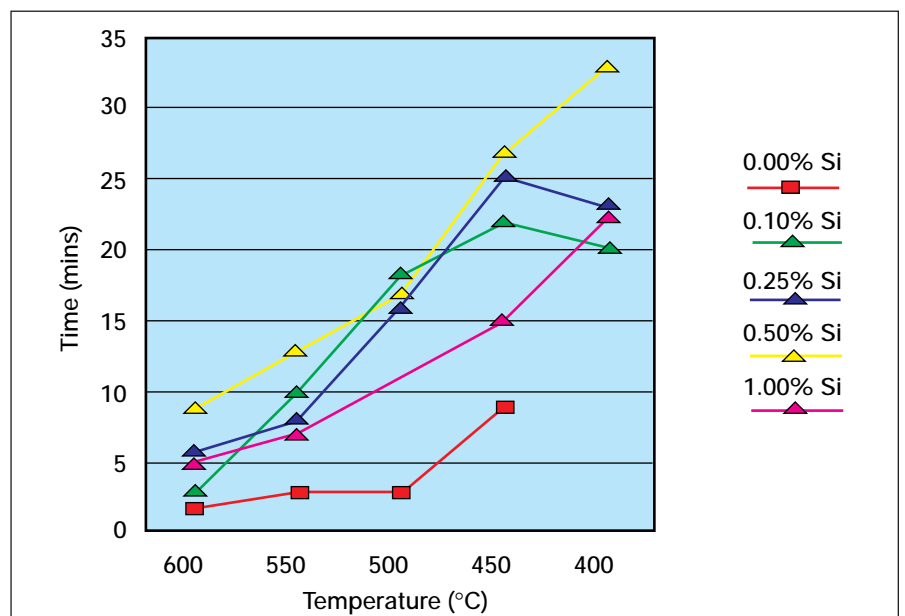


Figure 2 – Cooling Rate vs Temperature

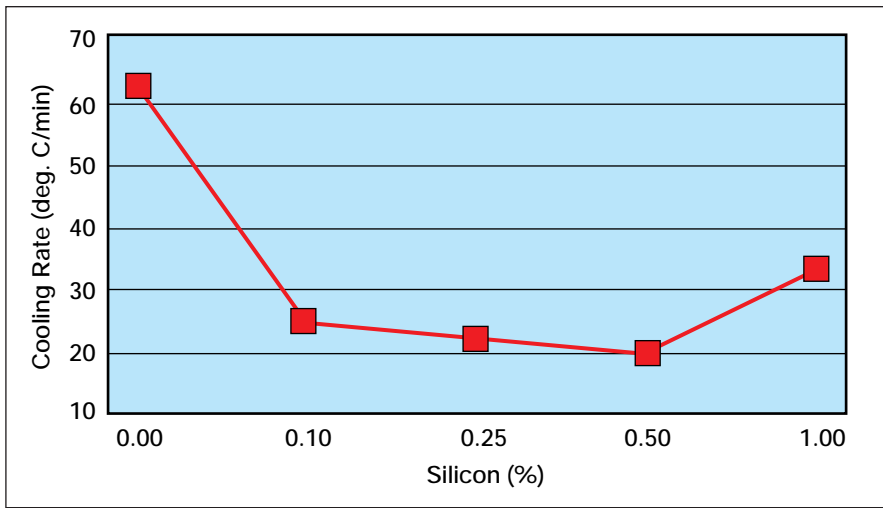


Figure 3 – Cooling Rate vs Silicon Content at 450°C

Theory states that the lower the temperature of a body, the slower the cooling rate. In these experiments the opposite was noted – the cooling rate decreased for the silicon-containing alloys even though they were at higher temperatures at any given time interval after casting compared to the silicon-free alloy. Some other factor must be influencing the cooling rate. It is suggested that the satin finish of the castings makes a significant contribution by lowering the emissivity – the amount of heat energy lost by a surface due to conduction, radiation, convection and the radiation received from surrounding bodies (10). Note that emissivity must not be confused with emissive power, which applies to radiated heat energy only. The increase noted for alloy 5 may be a result of the lower casting temperature used, a change in the emissivity due to the coarser, dendritic surface generated or a combination of both. The surface of alloy 1 was smooth but dull, again suggesting that the surface condition was affecting the cooling rate by raising the emissivity. Typical emissivity values quoted are:

- Lampblack – 1.00
- Dull copper – 0.79
- Polished copper – 0.48
- Polished silver – 0.02

It must also be noted that the rate of heat loss will also be complicated by shrinkage. If the alloy has a high thermal coefficient of expansion the shrinkage rate will be high, giving

rise to an air gap between the casting and investment. Below 600°C the rate of heat loss due to radiation is negligible compared to that of conduction. Air is a more efficient insulator than investment resulting in an overall decrease in cooling rate due to conduction and radiation effects. As noted earlier the cooling rate of an alloy will also be effected by it's specific heat capacity. Any increase in the specific heat capacity will result in a corresponding decrease in the cooling rate under

identical conditions since more heat energy must be dissipated per °C reduction. At the time of writing the variation of specific heat capacity between the alloys tested was not known and will require further work to establish.

This summation may be proved incorrect by future work; however based on the evidence available here it is a logical conclusion. It must be noted that whatever the reason behind the change in cooling rate, it is extremely significant. For a given time after casting to quench, the silicon containing alloys may have phase(s) present that remain in the liquid or semi-liquid state. Direct temperature measurement is essential; both sets of results indicate time is not a good indication for quench temperature.

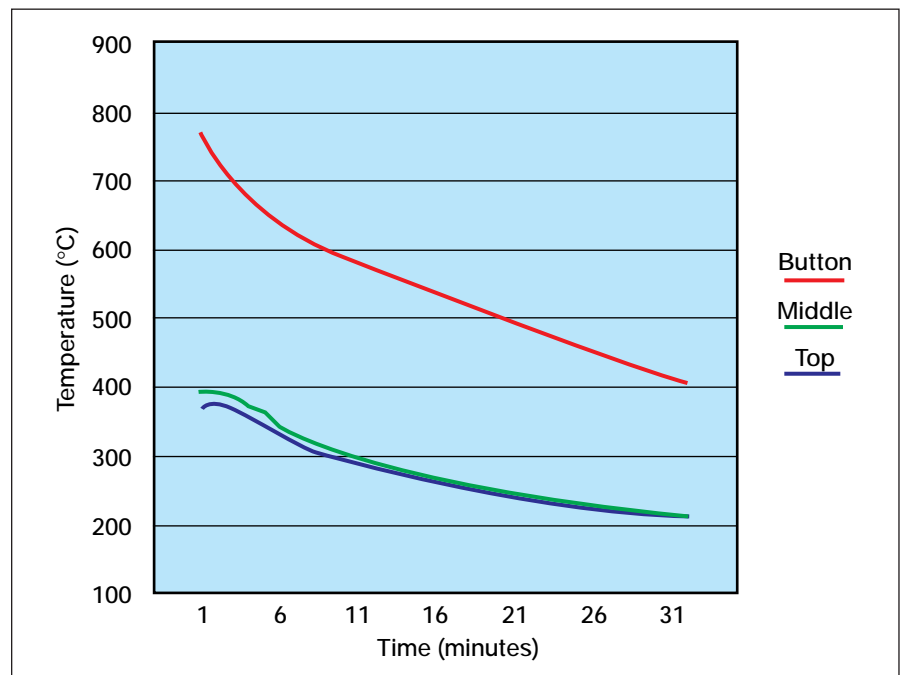


Figure 4 – Cooling Curves

Data taken from T1, T2 & T3 (button, middle and bottom of the tree) have been compared (figure 4), this being a typical cooling cycle. T1 was measured directly on the alloy surface, T2 & T3 assumed to be within a small distance of the castings. Even allowing for experimental error, it must be accepted that there is a decrease in temperature moving down the tree. The metal at the bottom of the tree (furthest from the button) was the first to come into contact with the relatively cold investment and will have cooled to a greater degree at any given time. The metal at the top (button end) of the tree will be the last to flow into the flask and therefore at a higher temperature as a result of superheat from the remainder of the tree and reduced time in the flask. The button will also experience a reduction in heat flow due to radiation from the open surface below 600°C decreasing significantly. The presence of low melting point phases would have a marked effect on the solidification characteristics of the alloy. In different tree positions at any given time, castings nearer the bottom half may have undergone full solidification whilst castings near the top half may still contain liquid or semi-liquid phases. As a result the potential for hot tearing will exist.

Time to temperature will be affected by the factors outlined above, but also by the size of tree, the charge weight and tree content. These will influence cooling rates drastically and may result in the problems noted when using high silicon alloys however the use of identical trees in this study has negated this effect.

### **Fluidity and surface finish**

All alloys filled the mesh pattern and ring settings completely indicating no measurable difference in fluidity from this test. It must be considered that differences may be noted if more demanding patterns were used; however since this study is primarily concerned with the effect of quench temperature on silicon addition and not silicon addition per se, no further work was carried out in this area.

The quenched and devested trees were subjected to an initial

examination prior to pickling. Only alloy 1 exhibited dark areas of copper oxide – all casting surfaces were grey/black. Alloys 2, 3 & 4 all exhibited a “satin” surface finish. Alloy 5 had an intermittent satin finish with “dull” areas however there were no indications of any oxides present. Trees were then pickled in a solution of sulphamic acid for 10 minutes, re-examined, and the amount of residual investment noted. For a given alloy, this increased with decreasing quench temperature, however this fact will be familiar to the investment caster and was expected. The amount of residual investment was also substantially higher for alloys 1 & 5 when compared with alloys 2 & 3, which were investment-free. Residual investment was present in minor quantities on the surface of alloy 4. This phenomenon is a direct consequence of the satin finish generated by the silicon additions being resistant to investment adherence.

Castings were then examined under the stereo microscope at x10 magnification. Alloy 1 exhibited a smooth but dull surface finish for all quench temperatures with evidence of dendritic formation being negligible. Alloys 2 & 3 exhibited a similarly smooth surface finish for all quench temperatures with evidence of dendritic formation again being negligible however the surfaces had a satin texture. Alloy 4 exhibited a smooth, satin surface finish for all quench temperatures; however evidence of dendritic formation was marginally increased over the previous alloys. The surface of alloy 5 exhibited a random dispersion of satin and dull textures for all quench temperatures. There was evidence of dendritic formation, this decreasing with decreasing quench temperature. Castings from this alloy exhibited the roughest surfaces of the study.

All alloys – apart from alloy 5 – were unaffected by quench temperature and gave similar results regardless of silicon content. The results for alloy 5 suggest a relationship between quench temperature and surface finish, with higher quench temperatures preventing filling of inter-dendritic spaces. It has been well documented

that higher silicon additions increase the potential for heavy dendritic growth (2,3). Since this alloy was introduced to the study as a control only and there are – to the best of the authors knowledge – no commercially available alloys with this level of silicon addition, no further investigation was carried out. For acceptable surface finish, the alloying limit may be considered as 0.5% silicon and the marginal decrease in surface quality accepted.

**Failure analysis**

For failure analysis, castings from tree design No. 1 were examined – these were actual work pieces and as such more relevant to the caster than tensile and wedge samples. Each tree contained 20 castings. Failure rates can be seen graphically (figure 5).

The failed castings from alloys 1 & 2 occurred at random positions on the trees. Metallographic examination confirmed these to be associated with areas of shrinkage porosity. For the remaining alloys, failure was predominantly at the button end of the tree, those with a high incidence having failures move down the tree with increased quench temperature. Only with alloy 5 quenched at 600°C were there failures at the end of the tree furthest from the button. It was also noted that failures in castings from the lower section of the trees or when few failures were identified were generally in the sprue area of the casting, i.e. the part expected to be hotter and last to solidify. This confirms a relationship between tree position and failure potential and suggests the theory regarding solidification and cooling rates was correct.

A high incidence of failure was predicted in alloy 5 when quenched at higher temperatures, however a definite trend existed suggesting a relationship between quench temperature and silicon content. Metallographic examination of the failures in the high silicon alloys confirmed the failure mechanism to be hot tearing of low melting point

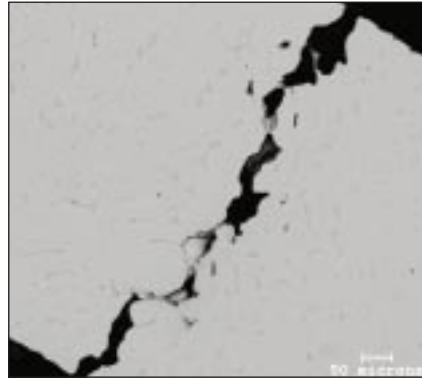


Figure 6 – Failure in alloy 4

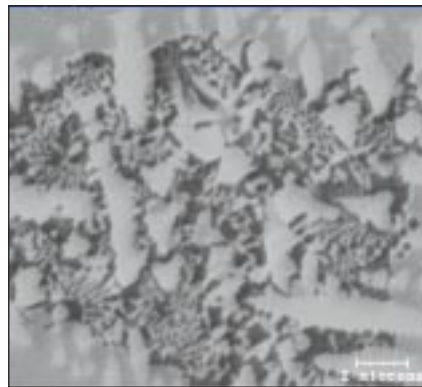


Figure 7 – Phase B

phases in conjunction with liquation pores and not brittle inter-granular failure (figure 6). Two phases were evident in micro section, one light and one dark – categorised as phases A & B respectively. Phase A was evident in all silicon containing alloys and was random in nature, increasing in intensity with increasing silicon content. Semi-quantitative analysis by

scanning electron microscope confirmed this to be a silver-rich, copper depleted silicon containing phase. There was no incidence of failure with phase A only present. Phase B (figure 7) was present in alloy 5 at all quench temperatures, but only marginal when quenched at 400°C. It was present in alloys 3 & 4 when quenched at 600°C & 500°C, but not at 400°C. The incidence of phase B increased with silicon content and would only precipitate within phase A. Semi-quantitative analysis revealed it to be a gold-rich phase with high levels of silicon. All failures by hot tearing were coincident with the presence of phase B. A silicon-rich grain boundary phase was identified at high magnification in alloys 3 & 4 and resulted in some minor grain boundary cracking; however no catastrophic failures were associated with this feature. Micrographs demonstrate the difference in appearance of the two phases during failure (figures 8 & 9). The smooth surface detected was phase B – confirmed by SEM analysis – with failure taking place also through the silver-rich phase A, which appears to be more tensile in nature.

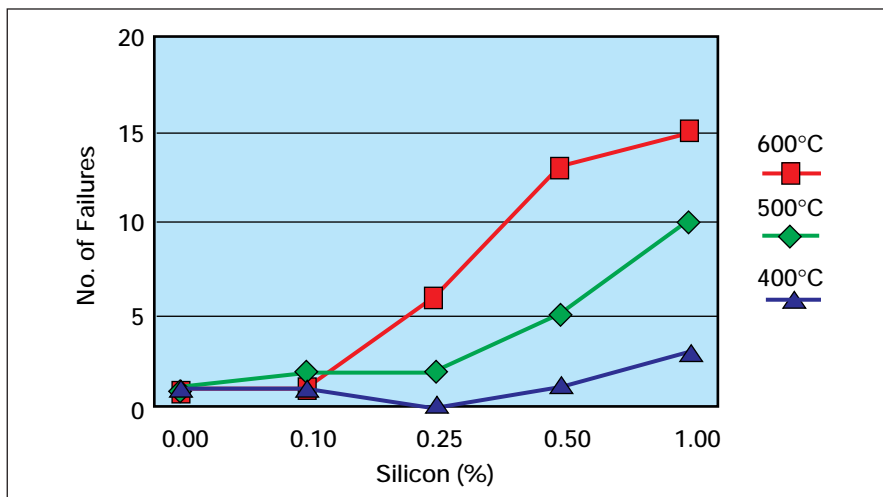


Figure 5 – Failure Rates vs Silicon



Figure 8 – Phase B on crack face

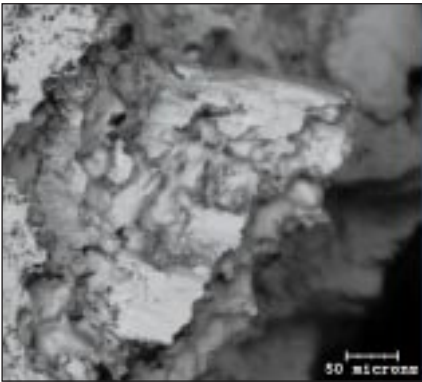


Figure 9 – Phase A & B on crack face

Examination of further castings confirmed that phase A was randomly dispersed throughout the matrix of all silicon containing alloys at all quench temperatures. Phase B was insoluble in high silicon alloys at elevated quench temperatures but soluble at lower levels and quench temperatures; it only existed as a duplex within phase A. At the time of writing neither phase had been positively identified.

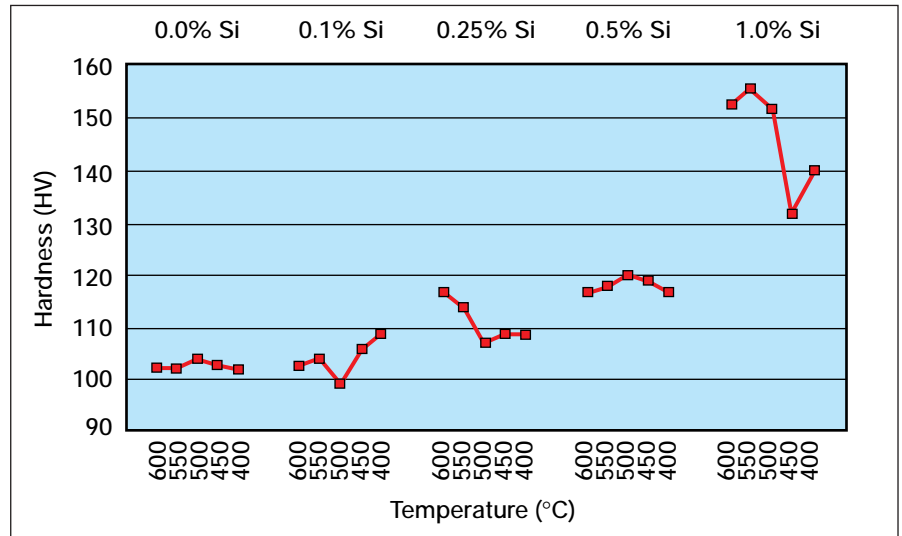


Figure 10 – Hardness vs Temperature

### Mechanical properties

The mechanical properties for each alloy as a function of quench temperature have been compared. From the data measured there appeared to be no correlation between the quench temperature and UTS, 0.2% proof stress, elongation and hardness. This was probably the result of the erratic nature of the segregates formed. Increasing the sample rate may yield data that is more representative and negate any anomalies. It has been observed in previous publications that an increase in silicon addition will change the mechanical properties, increasing the UTS, hardness etc. This was confirmed (figure 10); however for alloy 5 the elongation decreased considerably, fracture surfaces appearing brittle.

Quenching 9ct alloys above approximately 650°C results in the generation of a homogeneous solid solution  $\alpha$  (Au,Ag,Cu). The two phase region in the silver-copper binary results in the generation of an immiscibility curve below 650°C in the ternary system. If the alloy is slowly cooled below this temperature, the phases  $\alpha^1$ (Au-Ag) and  $\alpha^2$ (Au-Cu) will precipitate. This may be complicated further by the decomposition of  $\alpha^2$  into the ordered phase AuCu<sup>3</sup> – from the Au/Cu binary system (11,9) – resulting in some degree of hardening in the alloy. It was noted that the level of ( $\alpha^1 + \alpha^2$ ) precipitation increased with decreasing quench temperature as

expected; however the increase in silicon content also resulted in an increase in precipitation around the silicon-rich phases (figure 11 & 12). This may be related to the associated increase in cooling times allowing greater levels of precipitation and will require further investigation.



Figure 11 – Alloy 2 quenched at 400°C  
Magnification x 280

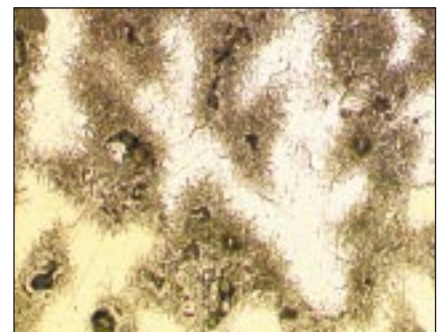


Figure 12 – Alloy 4 quenched at 400°C  
Magnification x 280

### Grain size analysis

No significant change in grain size as a function of quench temperature was noted for any of the alloys tested. Alloy 1 exhibited a minor increase from 0.8mm when quenched at 600°C to 1.0mm at 400°C; however this was not considered significant. The remaining alloys tested all exhibited similar average grain sizes of 2mm regardless of silicon content or quench temperature. All of the alloys tested here, in particular the silicon-containing alloys, would benefit from some degree of grain refinement. The generation of a fine grained cast structure would improve surface finish, corrosion and crack resistance. Grain refining may also influence the performance of these alloys during quenching – a finer grain structure may offer some degree of control on the otherwise erratic formation of the silicon-rich segregates identified, and as such improve quench strength.

### CONCLUSION

Analysis of the data generated in this study confirms susceptibility to failure during devesting increases with silicon addition for a given temperature. At levels of 0.25% silicon and above care must be taken when determining quench temperature to ensure complete solidification has been achieved. It was confirmed that the major mechanism of failure during devesting was not brittle fracture through grain boundary phases but hot tearing through low melting point silicon-rich phases. These were randomly dispersed throughout the alloy matrix.

The following factors were also determined:

- Low carat alloys may fall within the safe (Au+Ag) limit for a given silicon addition as determined by Normandeau & Roeterink and have acceptable post-casting properties but fail during devesting if a critical quench temperature is exceeded.
- The presence of the silicon-rich grain boundary phase often noted in higher carat alloys was negligible, precipitation in low carat alloys being into two randomly occurring irregular phases. Silicon additions of 0.1% and over precipitated a random irregular silicon containing silver-

rich phase. This increased in intensity with addition however was not affected by quench temperature. The presence of this phase alone was insufficient for failure to occur and evidence suggested it was ductile in nature. A second silicon-rich phase was identified under certain processing conditions. This was insoluble in the first at higher quench temperatures. It was the presence of this phase that resulted in the failures during devesting.

- The cooling rate of an alloy varied significantly with silicon content due to a number of factors outlined in the text.
- Time after casting was confirmed as an unreliable measure for quench temperature, direct measurement from the button being the most reliable indication. It was noted that this was not necessarily a true reading of temperature for all castings – this decreased moving away from the button. Calibration of cooling rate for high-silicon alloys may be required to establish the quench temperature limit below which failure will not occur.
- Mechanical properties as a function of quench temperature were erratic for all alloys. This was possibly a function of the nature of the silicon-containing phases formed. Determination of mechanical properties at elevated temperatures will yield further information regarding performance at various temperatures inside the mould cavity and during quenching. The hot strength of phases A&B will give an indication of the critical quench temperature above which failure will occur.

Overall, the use of alloys containing 0.5% silicon or less was determined to be acceptable provided certain production parameters were adhered to. A learning curve may be necessary when using high-silicon alloys enabling the caster to become familiar with an alloy and to have a measure – albeit subjective – for when the alloy has undergone complete solidification. There are several disadvantages when using high-silicon alloys however these are not catastrophic and their use is perfectly acceptable under the correct circumstances. It is suggested that a maximum level of 0.5% silicon be used

in low carat alloys – no advantage can be seen for any increased addition. If a high-silicon alloy is not specifically required it is suggested that one of lower content be used – 0.1% silicon offers a full range of beneficial properties with no disadvantages over the silicon-free alloy.

### ACKNOWLEDGEMENTS

The author would like to thank the following for their help in this study:

Peter Rotheram, Technical & Development Director, Cookson Precious Metals Ltd.

Bruce de Kocks.

Staff in the Metallurgical Department, Cookson Precious Metals Ltd.

Morgan Materials Technology Ltd. for the SEM, XRD and thermal analysis.

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