Confirming the cold diffusion bonding of gold in 19th century *kundan* settings

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ABSTRACT: This paper investigates the microstructural features of Indian gold settings know as kundan *using xenon plasma focused ion beam (pFIB), electron backscatter diffraction (EBSD) and energy dispersive x-ray spectroscopy (EDX). Testing sought to determine the purity of the gold foils in* kundan *settings and if they form diffusion bonds by cold-working. Three 19th-century samples of Indian* kundan *with reliable provenance were analysed. The samples were from the hilts of two Indian daggers given as diplomatic gifts to Albert Edward, Prince of Wales, during his tour of the Indian subcontinent in 1875-6. It was confirmed that diffusion bonding does occur in the cold-worked gold during the* kundan *process. Work hardening and mechanical condensing also contribute to the mechanical integrity of* kundan *settings. The settings were found to comprise of near pure gold foils of ~10 µm thickness.*

Introduction

Kundan is a term used to describe an Indian goldsmithing technique, best known as a method of setting gemstones in a high purity gold. The craft process involves mechanically crushing and compaction (also known as condensing in the dental literature) handmade gold foils around a gemstone and/or into a cavity. The initial seating of the first foil and the gemstone is often aided by a shellac-based adhesive. The foils are thought to form diffusion bonds from this work, undertaken at ambient temperatures with only the pressure of the craftsman's hand through the steel tools (Untracht 1997; Manuel 2004). Diffusion bonding of gold has been recorded in Vedic literature from approximately 1000 BCE (Dube 2018). However, as *kundan* is a unique cold working process and pre-sixteenth century examples are yet to be found, it must be evaluated separately. Compacted gold foil has also been used historically to fill dental cavities in multiple cultures. Although the first written accounts date from the sixteenth century, these are considered to draw on earlier Classical and Arabic medical practices (Gurunluoglu *et al.* 2003). The setting process of *kundan* has many parallels with the historic dental practice of foil fillings and with Vedic accounts of gold refining (Untracht 1997). Diffusion bonding in *kundan* settings has long been hypothesised but has not been confirmed (Untracht 1998; Manuel 2004; Stronge *et al.* 2015).

The Royal Collection contains many pieces of Indian decorative arts, particularly arms and armour, made using this technique. An understanding of the mechanisms behind this process and the resulting structure of the *kundan* is fundamental to creating conservation treatments for the care of such settings and related features. We wish to explore the validity of the claim that the foils used in *kundan* settings form a so-called self 'molecular bond' with minimal pressure, at ambient temperatures from near pure gold (Manuel 2004). Ambient temperature and low-pressure bonding of gold is interesting in this archaeometallurgical context but is also a pertinent area of study for microelectronics (Simons *et al.* 2000).

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Diffusion bonding is the interdiffusion of two materials encouraged by plastic deformation and often facilitated by heating (Tylecote 1978; Kazakov 1985). There have been various models proposed to describe diffusion bonding. The most prominent of these is the dislocation diffusion model where surfaces reduce their energy by dislocations coalescing (Kazakov 1985). Dislocations are defects in the crystal lattice of a material, they can be induced by stress such as bending or surface deformation (Scott 1991). One of the key challenges for diffusion bonding is surface cleanliness; native oxides and adsorbed hydrocarbon species on the surface of material can easily limit a material's ability to diffusion bond (Jellison 1975; Tylecote 1978; Kazakov 1985). For this reason, clean surfaces are required. Based on the dislocation diffusion model, a surface finish with a high dislocation density may facilitate enhanced bonding rates. The scratching or brushing of surfaces can be used to enhance the ability to form diffusion bonds by introducing dislocations and reducing oxide films and adsorbed species on the surface (Tylecote 1978).

Pure gold does not form a native oxide which is unique when compared to other less noble metals (Nutting and Nuttall 1977; Schmidbaur and Cihonski 2003). This absence of a native oxide is understood to allow both the beating/cold rolling of gold into submicron thickness and for diffusion bonding at ambient temperatures (Nutting and Nuttall 1977; Giumlia-Mair 2020). The absence of an oxide layer on gold is contingent on its purity, low carat (less than 75 %) gold may suffer from oxidation which would hinder bonding and processing to microns thickness (Schmidbaur and Cihonski 2003; Giumlia-Mair 2020). For future conservation work and to understand the bonding mechanisms, it will be necessary to characterise the composition of the gold used.

The thermomechanical history of the gold foils is also important when understanding the ability of the foils to diffusion bond. In the *Ain i Akbari*, a 16th century document recording the administration of the Mughal Empire under Emperor Akbar, the author Abū al-Faẕl writes a detailed account of the foil making process which it is worth sharing here in full:

> "of a masha [0.97 g] of gold they draw out a wire eight fingers long and one finger in breadth then the wire [sic] [The workpiece may take the form of wire or strip, given there is no description of a further deformation step] is coated with a mixture of two parts of the ashes of dried field-cow dung … and one part of Sambhar salt, after which it is wrapped in a coarse cloth and covered with clay. This is generally of not more than ten *tolahs* [116.6 g] weight and is placed in a fire of four *sers* [3.48 kg] of cow-dung which is then suffered to cool down. If there is but a little alloy in it, it will become of standard fineness after three fires, otherwise it must be coated with the same mixture and passed through three more fires. It is generally found that three coatings and three fires are sufficient for the purpose, it must then be washed and placed in an earthen vessel filled with lime juice or some other (acid) which is heated to boiling. It is then cleaned and wound round a cane and taken off (when required), and re-heated from time to time." (Abū al-Faẕl *et al.* 1891, 345–346).

The weights have been calculated using accepted conversion rates but are indicative rather than exact. The firing process described causes impurities in the gold to react with the salt and form chlorides. These are then absorbed by the clay or dissolved into the fruit acid once washed (Berger *et al.* 2021). This process of depletion gilding/cementation has been used in various forms since antiquity and could be repeated as many times as necessary to achieve complete purity (Abū al-Faẕl *et al.* 1891; Berger *et al.* 2021). Metallurgically, these steps would also anneal the foils after a deformation step (such as beating) to improve malleability. The use of dung ash is also mentioned in the Vedic literature referring to the cementation of gold (Dube 2018; Berger *et al.* 2021). The second round of heating is corroborated by contemporary craftspeople and echoes the dental literature where foils are passed through a flame before being worked into the setting or cavity. This de-gassing step removes any residual adsorbed species from the surface of the material to improve bonding (Knosp *et al.* 1981).

Having refined the foil, the substrate to which it will be applied must be prepared, whether this is a gold framework or an incised hardstone/animal tissue. In the case of a gold framework or 'ghat', the 'carcass' of the piece is similar to unfilled cloisonne cells (Stronge *et al.* 2015). These cells are filled with 'lac', a shellac-based mixture which is melted into the framework. The lac acts as both a filler and an adhesive into which foiled stones and/or the initial gold foil can be pressed. Once the stones and first foil are held by the cooled lac, the craftsman cuts pieces of gold foil off the pre-prepared roll of gold foil with snips, taking care not to touch them with bare hands, and passes them through a flame. Using pointed steel tools, the craftsman then crushes the foil around the stone with jabbing motions, crumpling and scrunching the foil into itself; the process is not dissimilar in action to needle felting. Once the foil or foils have been sufficiently crumpled, a small, chisel-like

Figure 1: A shows the T-shaped dagger (RCIN 11305). B is the nephrite hilted dagger (RCIN 11265). C is a macro image of kundan settings from the dagger RCIN 11305. D is a macro image of kundan settings of RCIN 11265 with missing fragments and residual lac in the setting. E is a fragment from RCIN 11305: the top cut face is shown. F shows a fragment from the same dagger with surface discolouration from creeping sulphide corrosion. G shows the top face of a sample from the dagger RCIN 11265. (A-B reproduced with permission from Royal Collection Trust / © His Majesty King Charles III, 2023).

tool, is 'walked' in a rocking motion over the area of compressed gold, to compact it. The zig-zag tool marks from this compressing can be seen on Figures 1C and 1D. All this 'work' results in one layer of gold. Three or four layers at least are needed to build up enough volume so that the compacted gold is around the same height as the stone. Once sufficient volume has been created, a flat graver is used to shave slivers of gold from the top surface to create a flat facet. Once the flat top has been established, the exterior and finally interior facets of the setting can be cut until the stone appears to sit in a nest of bright gold (Victoria and Albert Museum 2016; Marsolek 2023, pers. comm.).

Kundan can also be inlaid into a substrate in place of gold wire. However, if lac is not to be used, the host substrate must be prepared in the same way as for wire. Either an undercut or drilled holes must be cut into the design into which the foil can be forced and compacted to hold it in place.

Samples of *kundan* from the hilts of two Indian daggers have been studied to give insight into the process. The daggers were diplomatic gifts to Albert Edward, Prince of Wales, (later Edward VII) during his tour of the Indian subcontinent in 1875-6 and are now part of the Royal Collection.

Figure 1A shows RCIN 11305, an Indian dagger from the late 18th century with 19th century decoration. It is of *pesh-kabs* type with a recurved, crucible steel blade of strong 'T' section, tapering to a point with gold overlay decoration at the forte. The hilt is made of ivory scales, with rubies and diamonds *kundan* set in gold in a floral design. The hilt has a tassel of seed pearls and emerald beads, and the wooden scabbard is covered with fine panels of pierced and chased gold. Samples 1-2 were salvaged from this object when they became detached due to a mechanical failure in the setting. The channel cut into the ivory to receive the gold is very shallow and the drilled holes too dispersed to provide good anchor points for the gold.

The second dagger (Fig. 1B), RCIN 11265 is a 19th century Indian dagger of *khanjar* type with a double-edged, re-curved, crucible steel blade, partially overlaid in gold near the hilt (Fig. 1B). The hilt is formed from a single piece of carved nephrite jade. It is inlaid with diamonds, rubies and emeralds, also set in a floral design using *kundan* technique. It has an accompanying wooden scabbard covered with a deep purple silk velvet and fitted with a locket decorated in a similar manner to the hilt. The chased silver-gilt chape at the tip of the scabbard was probably a later addition. The dagger was presented by Jaswant Singh II, Maharaja of Jodhpur. Sample 3 was collected from this object in the same manner as those from RCIN 11305, though in this instance, the failure appears to have occurred due to an insufficient compacting of the gold surface, rather than improper preparation of the substrate.

The collected fragments were a consequence of mechanical failure of the settings. This failure and the visibility of toolmarks on the surface indicate the fragments are not of the highest quality manufacture. However, these visible features and their excellent provenance means they will provide an invaluable analogue for other objects from this era. Our work aims to understand the bonding mechanisms which underpin how *kundan* settings are formed and inform how such settings are conserved.

Analytical Methods

Following exploratory optical and metallographic investigation, analysis of the gold fragments was undertaken at the Loughborough Materials Characterisation Centre (LMCC) and the Hercules microcomputed X-ray tomography (micro-CT) centre. Three samples from the objects were characterised. The locations and orientation of each of the fragments is detailed in Figure 1. The fragments were initially documented with a Nikon SMZ25 macroscope.

Samples 2 and 3 were mounted in epoxy resin and polished with standard metallographic techniques. Where appropriate, the epoxy mounting materials were cut away for plasma Focused Ion Beam (pFIB) analysis. Carbon coating was used to make the epoxy coating conductive for scanning electron microscope (SEM) analysis while introducing little analytical interference to the elemental composition of the objects.

Plasma focused ion beam analysis (pFIB)

The layered structure, purity and low hardness of the compressed gold foils posed challenges for conventional metallographic preparation and etching (Chapman *et al.* 2021) and interpretation of compositional data may not be straightforward. Natural gold is rarely homogenous. Alloy heterogeneity is present as microfabrics formed either during primary mineralisation or by modification of pre-existing alloys by chemical and physical drivers during subsequent residence in either hypogene or surficial environments. Therefore, ion beam polishing was employed to expose and analyse the gold microstructure. Xenon ion milling was utilised for cross-sectioning large regions of the gold sample (> 300 μm). Ion milling involves the use of accelerated ions to sputter material away, allowing for highly site-specific processing with minimal mechanical damage. This method proved to be well-suited for the analysis of these gold fragments.

A Helios G4 Xe pFIB (ThermoFisher Scientific) was used for ion milling. The system was equipped with a 170 mm Utlim Max energy dispersive spectroscopy detector (EDS) (Oxford Instruments) and a symmetry3 electron backscatter detector (EBSD) (Oxford Instruments). Ion milling was conducted at 30 keV accelerating voltage and a range of currents appropriate to the cross-section width (2.5 μA – 60 nA) were used. Curtaining artefacts were observed in the larger cross sections which were cut at high current (2.5 μA). To reduce these artifacts XeF_2-assisted ion milling was used; the XeF_2 acts as a surface etchant, improving the surface finish of cuts. For the smaller cross sections (sample 3), platinum capping was used to reduce curtaining.

Scanning electron microscopy analysis (SEM)

Initial low magnification SEM analysis was collected with a Hitachi TM3030. Subsequent backscatter electron (BSE) micrographs were collected from the ion polished gold surface with a Helios G4 Xe pFIB. BSE images were collected at 5 keV and 1.6 nA. These images were used to measure foil thicknesses and identify microstructural features such as voids and twins. ImageJ was used to extract measurements from the BSE micrographs.

EDS was used to identify the composition of the gold and to identify any locally segregated elemental variations. An accelerating voltage of 30 keV and 1.6 nA was used, this ensured all the spectral lines of Au and Ag could be resolved. Each spectrum was collected to include 500,000 counts with a process time of 4 ms and dead time around 50 %. Al, Ti and Fe were omitted from the analysis as these elements were affected by scattered X-rays or spectral artifacts such as pulse pile up. Three spectra were collected from each sample to gain representative statistics. A gold standard wire (American Fine wire LTD 99.99% purity) was used as a reference material.

EBSD has been used to quantitatively characterise the grain size, grain orientations, grain boundary relationships and qualitatively assess the residual stress in the gold foils. Mapping was performed at 20–15 keV and 6.4–3.2 nA with a step size of 200 nm, 50 nm and 15 nm. In this research, we will quantify grains as regions with >10° misorientation.

Micro-CT

To better understand the internal structure of the gold fragments, non-destructive 3D X-ray computed tomography (CT) was carried out on a Zeiss Xradia CrystalCT. A beam voltage of 160 kV was used, and the specimen to source and specimen to detector distance was optimised, to allow analysis of the whole fragment, with a spatial resolution of approximately 1.2 µm. A filter was used to minimise artifacts. A total of 2401 projections were collected through a rotation span of 360°. Data collection took around 4 hours. The collected X-ray projections were reconstructed using Zeiss software, subsequent analysis was carried out in imageJ and Dragonfly 3D (Object Resource Systems).

Results and discussion

We will examine the samples from the macro to nano scale firstly assessing the surface topography.

Figure 1A–B shows the objects from which the three gold fragments were collected. The objects are impressive, but the settings are not of the highest quality (Fig. 1C–D). There are many loose tool marks and inconsistencies in the thickness of the settings. Figure 1C–D shows where fragments are missing. The fracture faces seem to be inclined suggesting the settings failed through their thickness, potentially where the original gold foils were intended to be compressed together.

The gold on the hilt of RCIN 11305 is set into a shallow channel. Small, drilled holes act as anchor points for the gold foil but with wider spacing and less depth compared to other similar pieces. As a result, the gold is poorly adhered and has sprung loose from its seating in places. In several areas, a darkening of the gold surface can be seen. This is probably caused by silver sulphide corrosion products that migrated from the silver foil, behind the gemstones, onto the gold surface (Fig. 1F). Sulphide creep is a well-documented corrosion phenomenon (Xu *et al.* 2010).

Figure 1E–G are optical images of the fragments. These posed challenges for optical microscopy as the surface is highly reflective. Optically, some tarnishing was observed on sample 2. As well as tarnishing, carbon and other contamination can be identified on the surfaces. To better understand the processing techniques, low magnification SEM analysis was conducted (Fig. 2).

Figure 2C shows that progressively filling the cavity with gold has replicated the drilled hole made in the hilt of the dagger. These holes are used to provide additional contact area to secure the *kundan* to the hilt. Due to the plastic nature of gold, the hole is very well reproduced without observable tearing or cracking, filling of a high aspect ratio hole with a less ductile metal would otherwise be challenging. Figure 2D shows the folded ends of sample 3, this image provides an indication of the lack of density in some areas of the *kundan*. We can also see that the gold foils are layered on top of each other in a consistent orientation and each foil has a similar thickness. While SEM analysis of the fragments was informative, limited information regarding the bonding or failure mechanism of the fragments was identified. To better understand the microstructure and internal density of the *kundan* samples, pFIB cross sections were made across the ends of the samples.

Figure 3 consists of low magnification backscatter electron images of the FIB cross-sectioned fragments. The dark area within these images indicates areas with low signal, revealing voids or areas of material with low atomic mass. The mottling within the foils and the contrast within the foils is indicative of channelling contrast, whereby different grain orientations allow the electron beam to penetrate into the material to a different degree, resulting in variation in signal. There is a significant variation in density between each of the samples. Cutting through Sample 1-2 (Fig. 3A–C), we

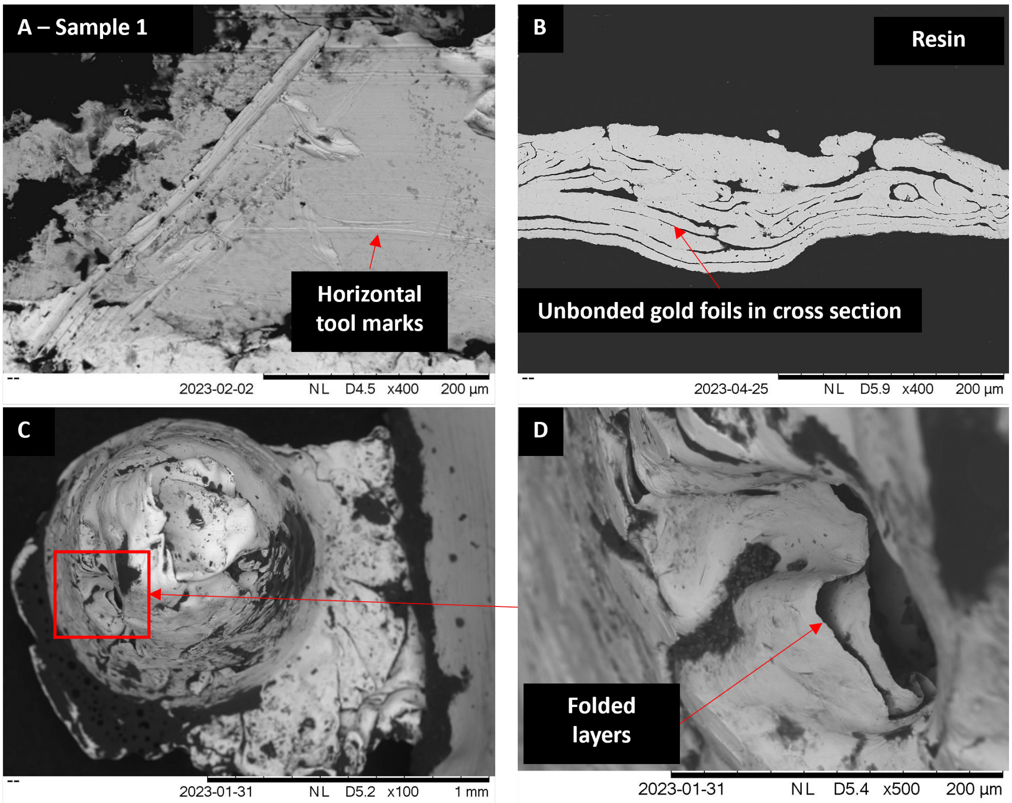

Figure 2: A. Low magnification SEM analysis of sample 11305F showing horizontal tool marks associated with the final finishing of the top surface. The oblique scratch is likely later damage. B. a mounted polished cross section image which shows the variation in density across the sample's thickness. C. low magnification image of the gold 'pimple' formed by pushing the gold foil into a drilled hole used for securing the kundan. D. higher magnification image of image C showing the ends of the foils which have been pushed together.

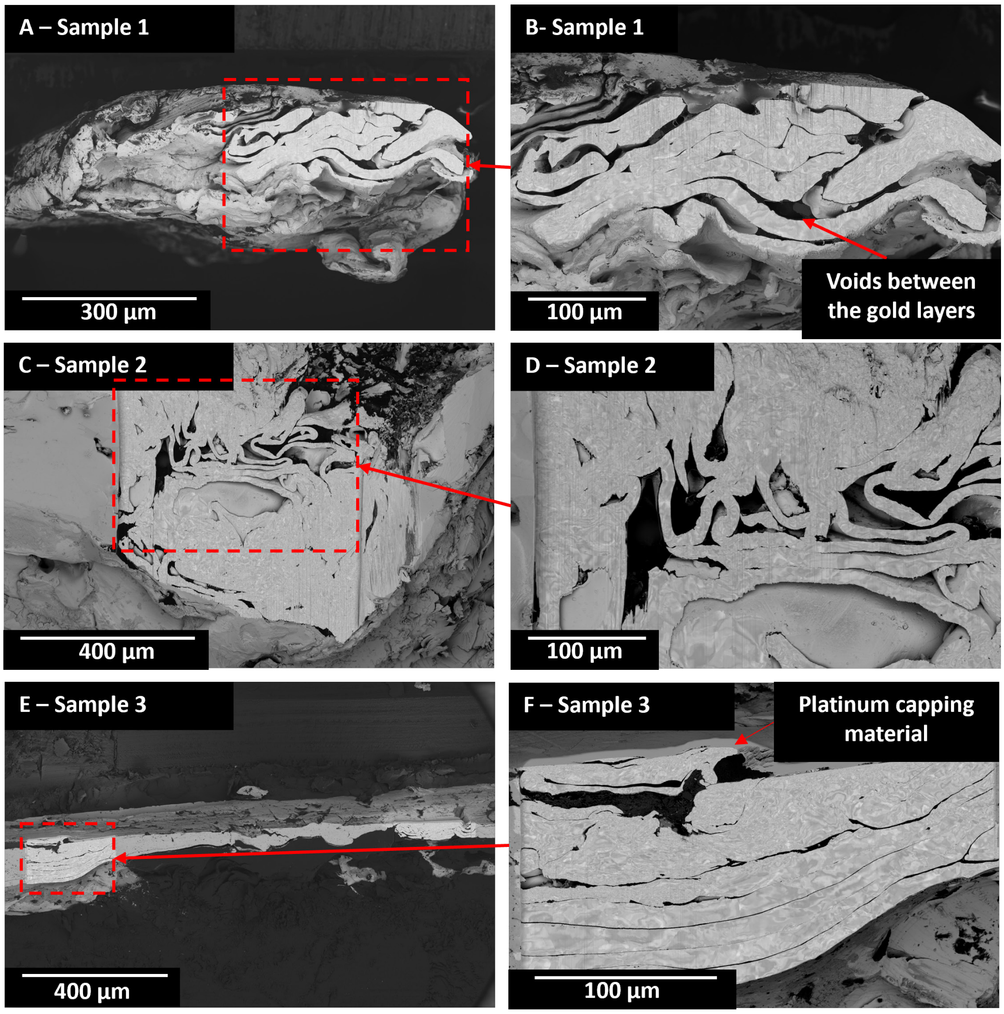

Figure 3: BSE images of pFIB cross sections from A, B. Sample 1. C, D. Sample 2. E,F. Sample 3.

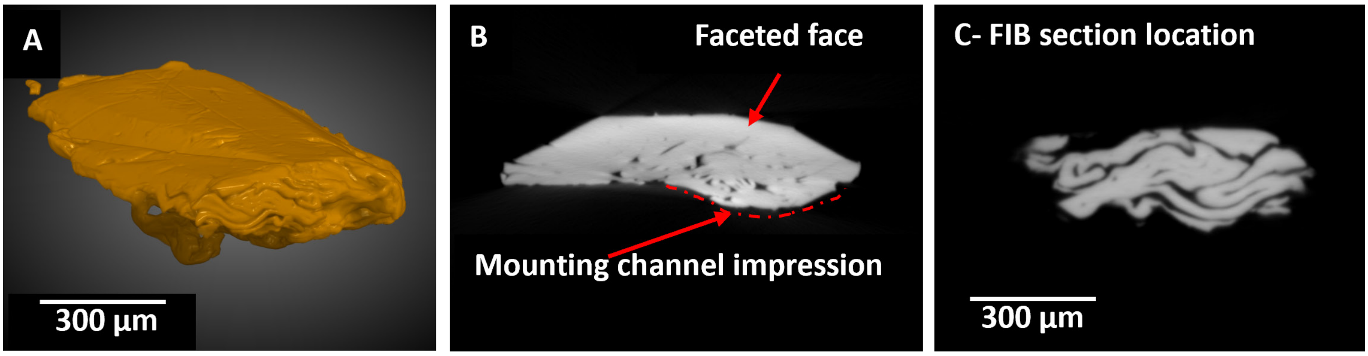

Figure 4: A. 3D rendering of sample 1. B. virtual cross section near the midline of the sample. C. cross section image of the pFIB milled region.

can see that the gold foils are not simply stacked and compressed. Rather, each piece of *kundan* is a complex folded structure while the structure is less complex and more simply layered for thinner sections such as sample 3 (Fig. 3E–F). It may be that craft people used density variation during the setting process to build volume in the settings quickly and to reduce cost.

Measurement of the foils in cross section indicates a thickness of 6.3–19 µm. This relatively small range suggests that craftspeople had a processing window for which gold foils are appropriate for *kundan* setting and demonstrates the high level of skill and precision required for this technique. The distinction between gold foils and leaf in the literature is that foils are self-supporting, typically greater than 10 µm in thickness and manufactured by rolling or beating (Darque-Ceretti *et al.* 2011; Giumlia-Mair 2020) while gold leaf is typically 0.1–1.2 µm thick and has undergone a further beating step. Beating is usually carried out with the leaves in a stack with an interleaving material, leaves are typically no longer self-supporting (Darque-Ceretti *et al.* 2011).

The cross-section of sample 1 was selected for detailed EBSD and BSE analysis. To confirm this location was representative of the bulk of the fragment, exploratory micro-CT data was collected.

From the micro-CT data (Fig. 4A), a better appreciation of the overall shape and internal structure of the setting can be gained. The top of the sample (Fig. 4B) forms a blunted triangle-like cross section, which would have been cut with a flat graver leaving the final polished finish of the setting, while the base of the setting (Fig. 4B) is less dense with witness marks where the gold would have sat in a mounting channel surrounded by lac. The cross section shows the chaotic nature of the folds. Figure 4C shows a virtual cross section close to the pFIB cross section location. The morphology of this area is similar to the cross section located midway through the fragment in Figure 4B. Micro-CT does not have sufficient resolution to resolve the micro and nano-scale features of diffusion bonds.

The EBSD analysis in Figure 5 allows us to resolve the grains of the *kundan* setting. What is evident from Figure 5 is foils in the undeformed state have a large grain size which spans the width of the foil with some grains ~100 µm in length. The large size of the grains in the undeformed region is indicative of a recrystallised microstructure (Ott and Raub 1981). Once the foil is cold worked, smaller grains form in the deformed material. This is particularly evident in areas where we see hairpin type folds (Fig. 5A). The kernel average misorientation maps (Fig. 5C) show that the folding and compaction of the foil are related to high degrees of misorientation in the grains. In the highly stressed zone, such as at the top of the EBSD map, we observed a fine grain zone 2.5–8.5 µm in thickness, likely from compaction and subsequent cutting of the facets on the top face of the foil (see the bottom of Fig. 5B). Cold working and reducing the grain size of the material would increase the material's hardness and allow for sharper finishing cuts to be made, resulting in a higher quality finish (Ott and Raub 1981).

Figure 6A shows locations with semi and well bonded regions. The EBSD analysis in Figure 6B can be correlated to compare the grain structure at these locations. What is observed is that fine grains are frequently associated to the well bonded regions and some of the semi-bonded regions. Two hairpin type bends are observed; grain recrystallisation can be seen on the interior and exterior faces of the bent regions. The hairpin bend at the bottom of the image has more significant grain refinement, suggesting more cold work. For densification and cold welding, the gold must have been compressed. This would require a continuous path between the substrate and tool. The faces between the void marked with a red cross (Fig. 6A–B) are mirrored, suggesting that at one stage these faces were joined. This observation may

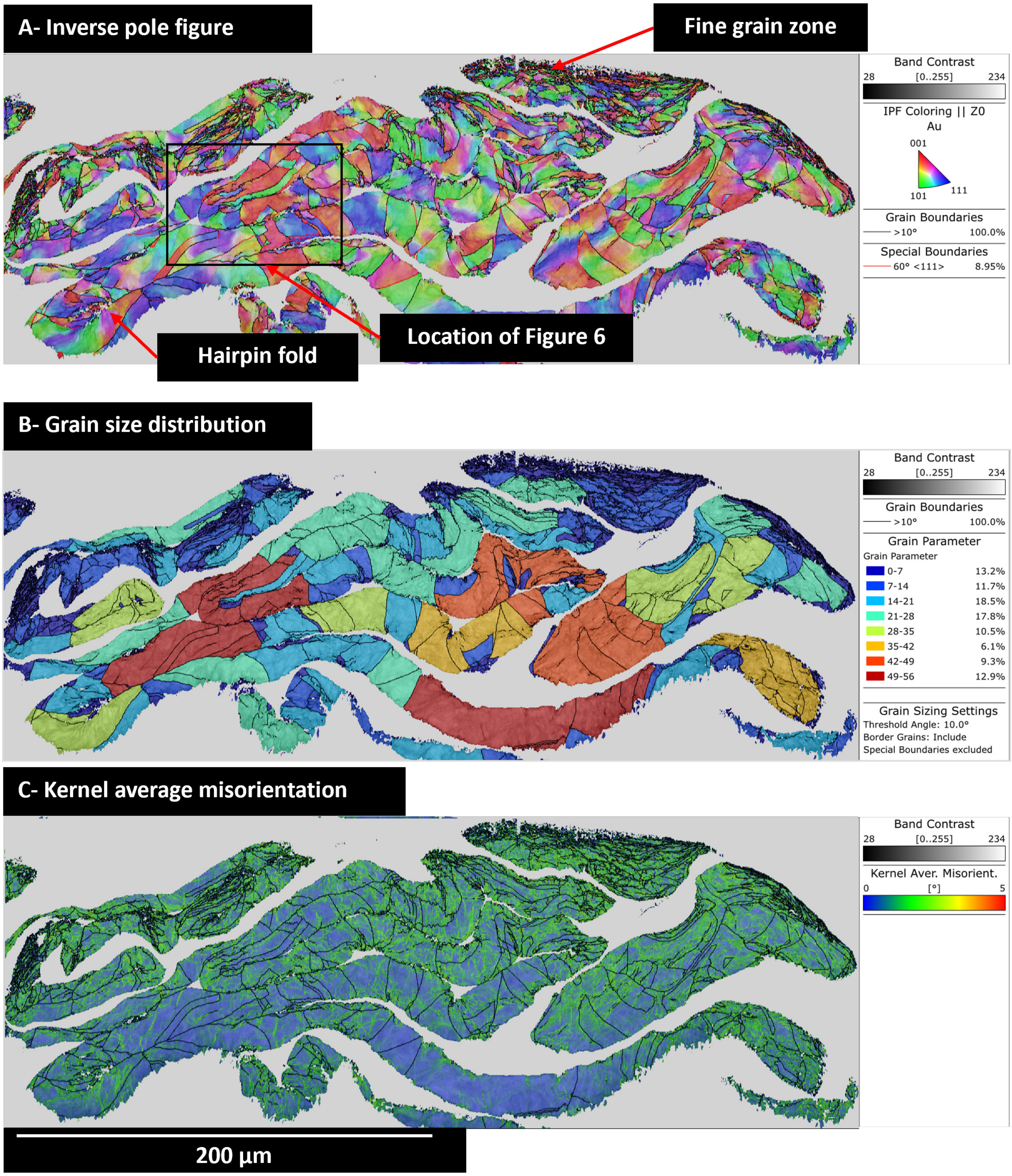

Figure 5: EBSD analysis of sample 1. A. Inverse pole figure and grain boundary map indicating the crystal orientation of each grain. B. Grain size map of equivalent circle diameter (ECD) with large grains in red and small grains in blue. C. Kernel average misorientation map where blue areas are low strain and green/yellow areas are higher strain.

explain the fine grains at the face of the two foils and the comparatively rough surface of the lower foil. These layers must have become delaminated during the objects working life or later handling and display. This suggests the diffusion bonded regions remain a point of weakness in the microstructure.

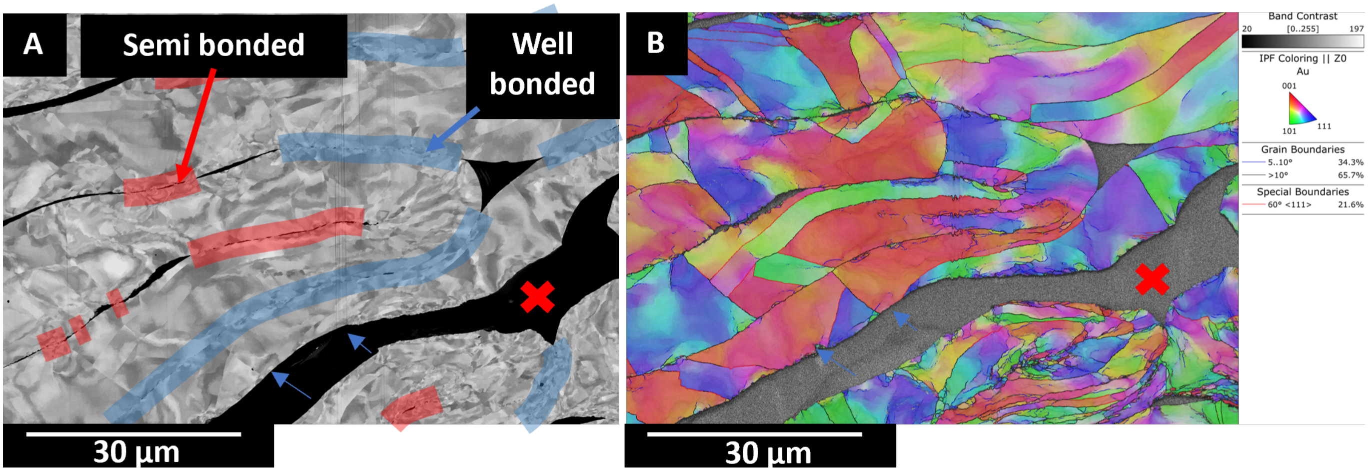

Figure 6: A. BSE image of Sample 1 with semi-bonded (red) and well-bonded zones (blue) indicated. B. EBSD inverse pole figure map from the area shown in A. The red cross and blue arrows denote the faces which we believe were previously bonded

Based on our observations, we have identified three characteristic types of diffusion bonds formed by *kundan* setting. These are semi-bonded zones, recrystallised semi-bonded zones and fully diffusion-bonded zones. In the semi-bonded regions, we see large grains at the interface and neck formations with widths on the order of 100 nm (Fig. 7A). The semi-bonded zones are frequently observed when the foils are stacked together with minimal cold work/bending (Fig. 6).

In the recrystallised semi-bonded regions, we see fine grains at the interface and many neck formations with voids trapped between necked regions, the necked formations are ~300 nm in width. The morphology of these neck features, elliptical voids and rounded voids, are consistent with the morphology of diffusion bonds reported in the literature (Shirzadi *et al.* 2001; Zhang *et al.* 2016). The literature examples of diffusion bonding are typified by high temperature and pressure bonding. However, these same features are evident at room temperature and low pressure in these settings, likely due to high ductility and inability of gold to form an oxide layer.

In the fully diffusion-bonded regions we observed a mix of elliptical and circular/rounded voids on the 20 nm scale. The bond line is no longer easily traced, and we see grains and twins spanning the interface. In the high magnification images, dislocations are visible (Fig. 7D). EBSD analysis shows that, despite the formation of fine grains, the parent foils maintain their original orientation and grain boundary misorientation greater than 10°. These findings suggest plastic-deformation induced solid-state diffusion has occurred. However, due to the low processing temperature of *kundan* setting, full recrystallisation of the bond line does not occur. This residual bond line results in a remaining weakness in the microstructure which could be an initiation point for failure if the settings were stressed.

The purity of the gold with which these bonds has been achieved will now be characterised. EDX point analysis was used to quantify the purity of the gold. The results were compared to a gold reference sample of a known purity (99.99 %).

Table 1 compares the elemental composition of the three samples and reference gold sample by EDX. Based on this analysis, the gold foils were around 97–99 % purity with Ag and Cu being the predominant impurities. Despite samples 1 and 2 originating from the same object, we observed differences in the compositions of the foils used. Silver and Cu impurities are consistent with those identified in the literature and are likely to reflect the craft foil preparation process (Cason *et al.* 2017; Craddock 2023). Microstructurally, we did not observe segregation or inclusions within the characterised foils. These findings support the claims that near pure gold is used (Untracht 1997; Manuel 2004). High purity gold is advantageous for this application as precipitates and alloying additions typically strengthen the gold matrix and limit the cold work which can be performed before cracking occurs (Simons *et al.* 2000; Cason *et al.* 2017). Further analysis using XRF or ICP-MS could be used to further quantify the composition of the samples (Craddock 2023).

Table 1: EDS analysis of gold samples in weight percentage (wt%). C, Ti, Fe and O omitted.

Label	Au	σ_{Au}	Ag	σ_{Ag}	Cu	σ_{Cu}	Total
Sample 1	99.0	0.06	0.9	0.05	0.1	0.02	100.0
Sample 2	99.9	0.03			0.1	0.03	100.0
Sample 3	97.2	0.11	2.4	0.12	0.4	0.02	100.0
Gold Standard	100.0						100.0

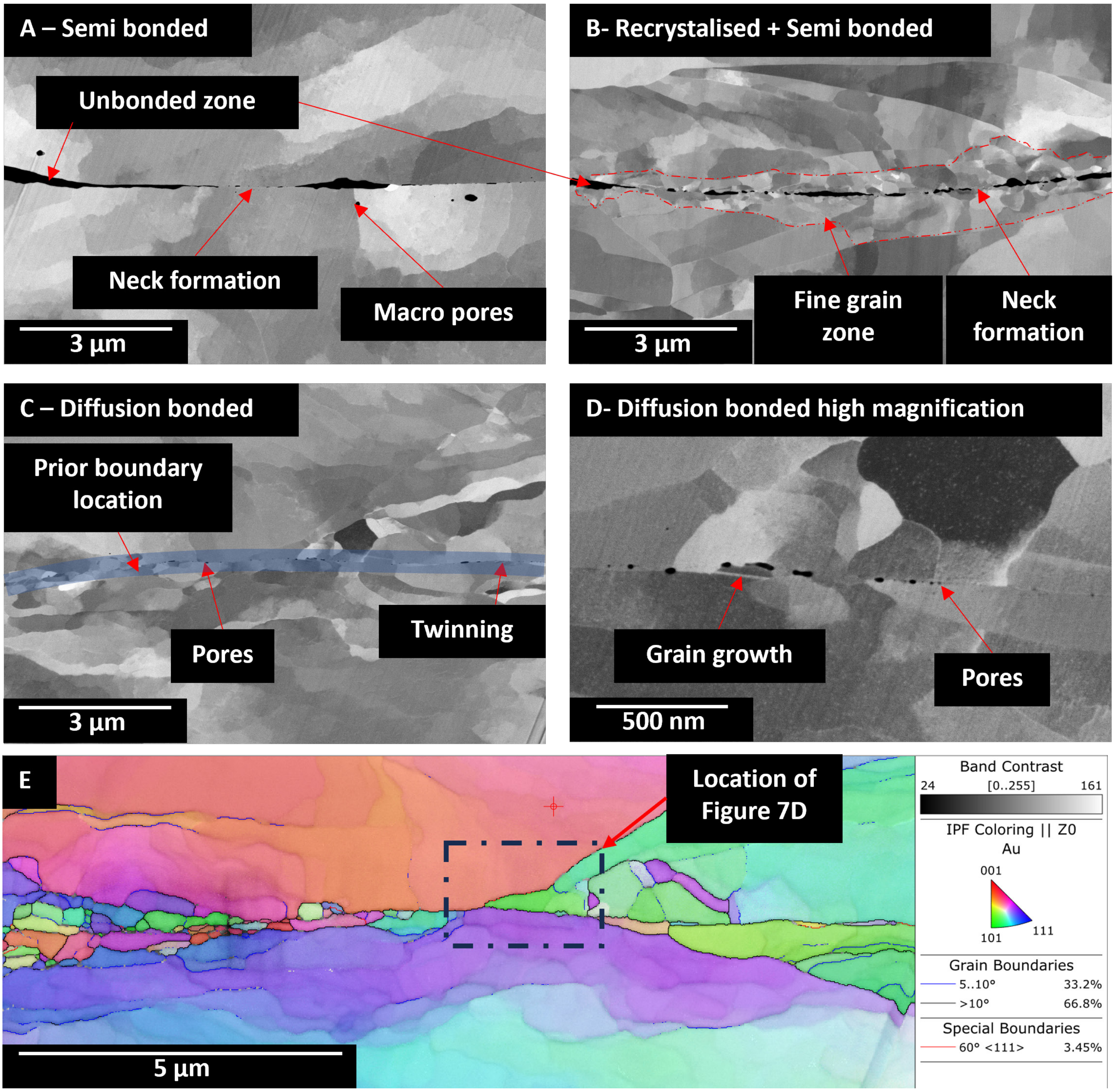

Figure 7: BSE and EBSD images of bonding locations within Figure 6. A shows a semi-bonded zone where a few contact points can be identifed. B shows a recrystallised and semi-bonded zone. C shows a well diffusion-bonded zone. D is a higher magnification image from image C. E. High magnification EBSD-IPF map of the bond line previously depicted in C-D. Note the amount of recrystallisation at the left hand end.

Conclusions

We have confirmed that the gold foils that make up *kundan* settings are of high purity gold, of above 97 % and do form diffusion bonds through cold working. Exploratory examination of the *kundan* fragments was carried out by SEM and conventional metallography. SEM images of the exterior of the fragments showed the folding of individual foils, variations in density and the replication of the form of the setting cavity. Cutting tool marks associated with the final finishing of the top surface were also revealed. Conventional metallographic sections confirmed dramatic differences in density within individual fragments but, because of the challenges associated with the reflectivity, preparation and etching of the gold used, we were unable to visualise the microstructure or any bonding. Consequently, micro-CT, plasma focused ion beam (pFIB) analysis and electron backscatter diffraction (EBSD) were used to characterise the internal microstructure, including grain orientation and size and, at higher magnifications, nanoscale features. Energy-dispersive X-ray spectroscopy (EDS) was used to determine composition.

We are confident to have clearly observed diffusion bonding in *kundan* settings. High magnification backscatter electron images distinctly show typical diffusion bonding microstructural features including neck formation, characteristic residual voids, large sections of consistent bonding and grain growth across the bond line interface. The number and size of bonds varied significantly from areas of the fragments where there were no bonds, through to areas where bonds were small and far apart, to other areas where bonds were continuous apart from the presence of nano-voids. Despite this evidence of bonding, the low temperatures and pressures used for bonding are insufficient to fully recrystallise large regions of the microstructure. Thus, the diffusion bonds would remain as a weakness in the microstructure.

Diffusion bonding is not the only mechanism which contributes to the mechanical integrity of *kundan* settings. The folding and crushing of the gold foils induces interlocking and work hardening which adds to the mechanical properties of the settings. The repeated folding and crushing of the foils produce leaf-spring-like characteristics which would also enhance the structural integrity of the setting. Based on the dislocation diffusion bonding model, we postulate that compressing and bending of the foils also contributes to the formation of the diffusion bonds, a theory which is also supported in the literature (Tylecote 1978).

With regards to the craft processes for refining and preparing the gold for bonding, the archaic methods described in texts such as the *Ain i Akbari* have utility and contribute to the outcome of the craft process. EDX found the gold, which was likely purified by cementation and pickling, was of a very high purity. Steps such as flame cleaning the foils before bonding have continued to be used in dental and commercial diffusion bonding applications to the modern day (Fletcher 1890; Jellison 1975; Kazakov 1985).

Further work will address the consequences of this understanding for the conservation of objects with *kundan* decoration.

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Bibliography

Abū al-Faẕl, ibn M., Blochmann, H. & Jarrett, S. (1891). *The Ain i Akbari*. Calcutta: The Asiatic Society of Bengal (Bibliotheca Indica).

Berger, D., Brauns, M., Brügmann, G., Pernicka, E., & Lockhoff, N. (2021). Revealing ancient gold parting with silver and copper isotopes: implications from cementation experiments and for the analysis of gold artefacts, *Archaeological and Anthropological Sciences* 13(9), 143. doi: 10.1007/s12520-021-01369-2

Cason, C., Pezzato, L., Brunelli, K., Furlan, F. & Dabalà, M. (2017). Effect of the composition and production process parameters on the microstructure, residual stresses, and mechanical and corrosion properties of gold alloys used in industrial jewelry processes, *Gold Bulletin* 50(3), 259–266. doi: 10.1007/s13404-017-0215-y

Chapman, R. J., Banks, D. A., Styles, M. T., Walshaw, R. D., Piazolo, S., Morgan, D. J., Grimshaw, M. R., Spence-Jones, C. P., Matthews, T. J. & Borovinskaya, O. (2021). Chemical and physical heterogeneity within native gold: implications for the design of gold particle studies, *Mineralium Deposita* 56(8), 1563–1588. doi: 10.1007/s00126-020-01036-x

Craddock, P. T. (2023). The assay and refining of gold in the post-medieval Islamic world: Potential and reality, *Historical Metallurgy* 54, 9–20. doi: 10.54841/hm.661

Darque-Ceretti, E., Felder, E. & Aucouturier, M. (2011). Foil and leaf gilding on cultural artifacts: forming and adhesion, *Matéria (Rio de Janeiro)* 16(1), 540–559. doi: 10.1590/S1517-70762011000100002

Dube, R. K. (2018). Solid state diffusion bonding of gold: a Sanskrit reference from Vedic literature, *Historical Metallurgy* 52(2). Retrieved 26 April 2025, from: https://hmsjournal.org/index.php/home/article/view/28.

Fletcher, T. (1890). *Practical dental metallurgy*. Warrington: Mackie and Co Limited.

Giumlia-Mair, A. (2020). Plating and Surface Treatments on Ancient Metalwork, *Advances in Archaeomaterials* 1(1), 1–26. doi: 10.1016/j.aia.2020.10.001

Gurunluoglu, R., Gurunluoglu, A. & Piza-Katzer, H. (2003). Review of the "Chirurgia" of Giovanni de Vigo: Estimate of his Position in the History of Surgery, *World Journal of Surgery* 27(5), 616–623. doi: 10.1007/s00268-003-6819-9

Jellison, J. (1975). Effect of Surface Contamination on the Thermocompression Bondability of Gold, *IEEE Transactions on Parts, Hybrids, and Packaging* 11(3), 206–211. doi: 10.1109/TPHP.1975.1135065

Kazakov, N. F. (1985). A Theory of Diffusion Bonding. In Kazakov, N. F. (Ed.) *Diffusion Bonding of Materials* (pp. 17–48). Oxford: Pergamon. doi: 10.1016/B978-0-08-032550-7.50006-0

Knosp, H., Nawaz, M. & Stümke, M. (1981). Dental gold alloys: Composition, properties and applications, *Gold Bulletin* 14(2), 57–64. doi: 10.1007/BF03214598

Manuel, K. (2004). The Kundan Technique: The Indian Jewellers Unique Artistic Treasure. In Crill, R, Topsfield, A. & Stronge, S. (Eds.) *Arts of Mughal India: Studies in Honour of Robert Skelton*. London: Victoria and Albert Museum.

Nutting, J. & Nuttall, J. L. (1977). The malleability of gold: An explanation of its unique mode of deformation, *Gold Bulletin* 10(1), 2–8. doi: 10.1007/BF03216517

Ott, D. & Raub, C. J. (1981). Grain size of gold and gold alloys: A review and some recent developments, *Gold Bulletin* 14(2), 69–74. doi: 10.1007/BF03214600

Schmidbaur, H. & Cihonski, J. L. (2003). Noble Metals (Chemistry). In Meyers, R. A. (Ed.) *Encyclopedia of Physical Science and Technology* (3rd edition, pp. 463–492). Amsterdam: Elsevier. doi: 10.1016/B0-12-227410-5/00481-6

Scott, D. A. (1991). *Metallography and Microstructure of Ancient and Historic Metals*. Los Angeles: The Getty Conservation Institute. Retrieved 26 April 2025 from: https://www.getty.edu/conservation/publications_resources/pdf_publications/pdf/metallography.pdf.

Shirzadi, A. A., Assadi, H. & Wallach, E. R. (2001). Interface evolution and bond strength when diffusion bonding materials with stable oxide films, *Surface and Interface Analysis* 31(7), 609–618. doi: 10.1002/sia.1088

Simons, C., Schräpler, L. & Herklotz, G. (2000). Doped and low-alloyed gold bonding wires, *Gold Bulletin* 33(3), 89–96. doi: 10.1007/BF03215484

Stronge, S., Whalley, J. & Ferrari, A. (2015). *Bejewelled Treasures: The Al Thani Collection*. London: V&A Publishing.

Tylecote, R. F. (1978). The solid phase bonding of gold to metals: Historical and more fundamental aspects, *Gold Bulletin* 11(3), 74–80. doi: 10.1007/BF03215091

Untracht, O. (1997). *Traditional Jewelry of India*. London: Thames and Hudson.

Untracht, O. (1998). *Jewelry Concepts and Technology*. London: Robert Hale.

Victoria and Albert Museum (2016). *How Was It Made? Crafting a Pair of Traditional Enamelled Earrings with Kundan Set Diamonds* [Video]. YouTube, https://www.youtube.com/watch?v=ZLvYOTaHEZM

Xu, C., Reents, W., Franey, J., Yaemsiri, J. & Devaney, J. (2010). Creep corrosion of OSP and ImAg PWB finishes. *SMT Surface Mount Technology Magazine* 25, 12–25.

Zhang, C., Li, H. & Li, M. (2016). Detailed Evolution Mechanism of Interfacial Void Morphology in Diffusion Bonding, *Journal of Materials Science & Technology* 32(3), 259–264. doi: 10.1016/j.jmst.2015.12.002

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