**REPORT TO:** 

### Stained Glass Resources, Inc. Hampden, MA

Attn: Mr. Frederick B. Shea

# Analysis and Comparison of Used and New Lead Window Cames

MMR Project No. 0463-21-1

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From Massachusetts Materials Research, Inc.

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#### BACKGROUND

Stained Glass Resources, Inc. of Hampden, Massachusetts requested that Massachusetts Materials Research, Inc. (MMR), West Boylston, Massachusetts compare and evaluate the lead cames from various sources and document any age-related differences that may exist. A representative of MMR visited the Stained Glass Resources facility to observe the various steps involved in the releading process. An overview of the common industry practice of window repair was also provided during this visit. This overview included pressing a window flat again and resoldering cames.

As a result of this visit and the information provided, MMR developed a testing and evaluation plan that included the following:

- Binocular microscope examination of the came samples,
- Scanning electron microscope (SEM) examination of fracture surfaces present on old cames,
- Energy dispersive x-ray spectroscopy (EDS) analysis of fracture surfaces present on old cames,
- Comparative tensile testing of old and new cames, and
- Metallurgical analysis of old and new joints and old and new cames.

These analyses were chosen as the best ways to present any differences noted between the old, used cames and new cames, and to evaluate these differences with respect to the structural integrity of a window. The term "old" as used in this report refers to cames produced from the mid-nineteenth to mid-twentieth centuries. The lead cames provide the structural framework to support the glass panes, much as a frame provides support to a canvas. Documenting the effects of time, stress, and atmospheric exposure, as well as differences between repairs and replacement product can help develop a more scientific way to evaluate window conditions.

#### **RESULTS OF TECHNICAL INVESTIGATION**

#### Visual Examination

Old cames from two windows were chosen for extensive evaluation with respect to new cames. Resoldered joints from a repair performed in the mid-1970's were chosen for evaluation with respect to a new joint. Resoldering cracks and joints is reportedly a widespread practice in window repair. Therefore, comparison of the joints produced is key when evaluating the effects of repairing versus releading. The samples chosen for testing were provided by Stained Glass Resources but selected by MMR. These samples are described below.

Sample	Date	Туре
А	~ 1913	came and joint
В	~ 1930	came
С	new	came
D	new	came
E	new	joint
F	~ 1970's	resoldered joint
G	~ 1970's	resoldered joint

## Table ILead Samples for Analysis

Visual examination of the older cames revealed a multitude of fine cracks extending into the came from its outer edges. Figure 1 shows several of these cracks along a one-inch length of Sample A. The older joints revealed widespread cracking as well. Figures 2 and 3 show joint cracks in Sample A window joints. The crack in the lower joint shown in Figure 2 was later examined with EDS analysis. For comparison, Sample E, a new joint with new cames is shown in Figure 4. Visible cracks were common to all older samples and not present on new samples. Since the cracking visible with the naked eye is not necessarily the only cracking present, further microscopic examination was performed.

#### **Binocular Microscope Examination**

A binocular microscope is a light microscope of the type commonly pictured when the word "microscope" is mentioned. Another term for this piece of equipment is stereo microscope.

This examination was conducted to allow inspection of the subject cames at magnifications up to 50X. Selected cracks were carefully broken open to reveal their fracture surfaces and examined with this method as well.

This examination did not reveal any new information with regard to the came surfaces. The fracture surfaces, however, were obviously different in appearance from the bright, shiny laboratory-created surfaces formed upon exposing the cracks. When a fracture is opened for inspection, metal that was still intact nearby the crack in questions produces a new fracture. This is the laboratory-created fracture. While it is not related to the initial crack, it can provide information about the base metal to compare with the crack in question. Figures 5 and 6 show the fracture surfaces of cracks in the came Samples A and B, respectively. Both photographs were taken with the same settings under the same lighting conditions within minutes of each other. Note that the fracture surface of the Sample A crack is noticeably darker than that of the Sample B crack. A portion of the laboratory-created fracture is visible in Figure 6. This laboratory-created crack is knife-edged and shiny. Contrast this bright, shiny appearance with the older fracture surfaces. The darker fracture surfaces are likely the result of greater oxidation. To verify that greater oxidation is the cause of the difference in appearance, these fracture surfaces were examined in a scanning electron microscope.

#### Scanning Electron Microscope (SEM) Analysis

Scanning electron microscope, or SEM, analysis was used for two reasons in this investigation: to reveal crack fracture mode, if not too heavily corroded, and to analyze the surface for differing oxygen levels to see if there was a detectable difference between samples of different ages. A SEM is different from a binocular microscope in that it uses an electron beam instead of light to form an image of the surface being analyzed. This means that the resolution and depth-of-field is greatly increased. SEM analysis provides for viewing of samples at much higher magnification than binocular microscopes.

The surfaces of the cracks shown in Figures 5 and 6 were examined in both the asreceived and cleaned conditions. The oxide layer present on both surfaces obscured the fracture features in the as-received condition, so a light cleaning solution of a substance known as Alconox was used to remove it. After cleaning, both fracture surfaces exhibited ductile dimple rupture fracture mode with extensive stretching and tearing, Figure 7. This indicates a very ductile, or deformable, metal. This is the same fracture mode that most ductile metals exhibit under tensile testing except that the test specimens typically lack the tearing features. It represents exposure of the metal to a force beyond its physical capabilities to withstand. Such tearing could occur from unusually high wind gusts, undersized cames, lead creep, out-of-alignment panes, or the weight of the glass over time.

The flat surfaces of the Sample A came were also examined to check for cracks not visible to the naked eye. Several randomly-selected regions were examined and approximately one third of them possessed a crack. Several of these cracks are shown in Figures 8 through 10. Note that the magnifications in figures range from 50X to 500X. None of these cracks was visible to the naked eye and only one was visible at 15X (shown in Figure 10 at 50X for greater clarity). Figure 11 shows the region where the crack pictured in Figure 8 was located. Note that it is not visible at 15X. This means that any repairs carried out on visible cracks leave a multitude of cracks untouched and unremedied.

The oxide layer itself and any differences that might exist between it on cames of different ages was also examined. This examination occurred prior to cleaning. To analyze this, energy dispersive x-ray spectroscopy, or EDS, was used to analyze the two fracture surfaces in question along with baseline laboratory-created fracture surfaces. EDS analysis uses equipment attached to a SEM to reveal the elements present in the analyzed region based upon characteristic x-ray emissions from the specimen. This is a qualitative microchemical analysis technique, meaning it detects relative amounts of elements. It cannot detect compounds (i.e. it will detect sodium and chlorine, but not sodium chloride) or determine percent composition. It will produce graphs, called spectrograms, that show peaks of various heights that correspond to an element's relative abundance in the analyzed region. In this way, it becomes easy to see in a graphical manner which region possesses more oxygen.

Figures 12 and 13 are the spectrograms for Sample A old fracture (present upon sample receipt) and new fracture (laboratory created). The difference in oxygen levels is readily apparent with the old fracture possessing an oxygen peak approximately three times as high as the laboratory created fracture.

The difference is a little less striking in Figures 14 and 15, which show the old and laboratory-created fracture oxygen levels of Sample B. The old fracture oxygen peak is approximately half again as high as the new fracture peak. Recall that Sample B is younger than Sample A, so age-related cracking would likely occur later in Sample B, assuming similarity of stresses and environment. This translates into less oxidation time for the Sample B crack than for the Sample A crack.

Oxidation produces a layer of corrosion product on the surface of a crack. As time passes, this layer becomes thicker as more metal is consumed by the corrosion process. To evaluate the thickness of this layer, metallurgical mounts were created.

#### **Metallurgical Analysis**

Several samples were mounted in clear epoxy and ground and polished to reveal the interiors of soldered joints and profiles of cames. These resulting "mounts" were examined in the as-polished condition to provide for the best contrast between solder and came metal and any cracks, voids, or inclusions present.

Figure 16 shows a new solder joint, Sample E, created for comparison. The cames joined by the solder are marked "C1" and "C2", and the solder is marked with an "S". Note that there are no gaps between the cames and the solder and the solder is solid with no inclusions (i.e. foreign particles), cracks, porosity (i.e. holes), or regions with lack of fusion. This was consistent along the entire joint.

Figure 17 shows a joint, Sample F, that was resoldered in the mid-1970's. Note the dark round shapes indicative of porosity and how the new solder appears from the OD to join a much larger amount of metal than it actually does. At higher magnification, the extent of the lack of fusion is revealed to be even greater than it originally appeared in the lower magnification view, Figure 18. Large regions of porosity and lack of fusion such as this should not be present in a structural joint. The smooth profile of the new joint and solid fill of its solder provides a joint of greater soundness than the material of the resoldered joint. Porosity and lack of fusion represent discrete regions where gaps in the joint exist. The jagged profile of the joint creates sites known as "stress raisers", or places where the stresses the joint experiences are magnified due to geometry. Stress raisers can accelerate joint failure.

Metallurgical mounts also reveal the depth of any oxide layer present. Figures 19 through 21 show the profiles of the came walls of the new sample, Sample C, and of older cames Samples A and B, respectively. As expected, the new came, shown in Figure 19, possesses no visible oxide layer. Sample A, Figure 20, possesses a well-developed, tightly adhered oxide layer on the came OD. Debris visible on the came ID is caulking remnant. The oxide layer is approximately 0.008-inch thick. Lead is known to produce a protective oxide layer, so this very thin layer is expected and normal, even after approximately 91 years of exposure to air.

Sample B, dating from the 1930's, is shown in Figure 21. The oxide layer present on this sample is approximately 0.0005-inch thick. The thickness difference is negligible and the non-continuous layer of Sample B was very likely caused by oxide spalling, or falling off, during removal from its window.

In summary, metallurgical examination revealed negligible oxide layer differences between the two older samples studied and a new sample. This is normal as lead is known to produce an adherent, protective oxide layer when exposed to the elements. Once formed, a protective oxide layer greatly decreases further oxidation, and a relatively stable condition is achieved.

What this examination also revealed was a notable difference between a new joint and an older, resoldered joint. The new joint was solid, lacked porosity, and was well fused to the cames. The resoldered joint possessed porosity, lack of fusion, a jagged stress-raising profile, and spotty fusion to a came. All these make the resoldered joint a much weaker construct.

The philosophy behind resoldered joints or repair soldering of cracked cames considers the resulting joints "good as new" if done "properly". Properly generally refers to adequate cleaning, temperature control, flux selection, and joint design. However, as this and SEM examination showed, came cracks possess a layer of oxidation. No matter how well the flat came surface is scrubbed or cleaned, the crack fracture surface oxide layer, due to geometry, will persist. Fluxes are not substitutes for cleaning and cannot remove such persistent, well-adhered oxide layers. They should not be counted on to do so. Fluxes remove tarnish films from precleaned surfaces, prevent oxidation during the soldering process, and lower the surface tension of the solder. Soldering over an oxidefilled crack will not produce a bond that is metallurgically equivalent to a new, uncracked length of came. It may even produce undesirable brittle intermetallic compounds in and near the soldered joint that accelerate cracking of the joint.

As Figure 3 shows, cracking at resoldered joints is a concern. In addition to the crack, note the jagged came form and melt-through regions at this T-joint. These are all hallmarks of a poor resoldering. The melt-through and jagged eaten-away appearance of the came results from too high heat and/or too long a contact between the soldering tool and the came in these regions. All the stress raiser issues previously discussed regarding uneven geometry are illustrated here. Cracks in the weld toe region, common in the samples examined here from different windows, are the result of the metal attempting to accommodate strains induced by the soldering. This can be due to excessive heat application, entrapped flux, creation of brittle intermetallic compounds, or poor stress distribution elsewhere along the came due to other repair work.

The prominence of such cracks in the samples examined from different sources suggests that they are less the result of the skill level of the person resoldering the joint (although the overall quality of the Figure 3 joint is very low) than of the difficulty in properly cleaning and designing a repair joint when the approach is to resolder and call it good.

Also, as noted in the SEM examination section, the visible cracks are not the only cracks present on a came. Many of the cracks present on the came surfaces examined were visible only at magnifications over 100X. Locating all such cracks on a sample intended for repair would require extensive microscopic examination.

#### **Tensile Testing**

Tensile testing was performed on samples of older cames and samples of new cames. Tensile testing was chosen as a test for this evaluation because it can provide an at-aglance comparison between specimens. This type of testing pulls a specimen in tension at a slow, controlled rate until the specimen ruptures, or breaks. The sample cames, both old and new, were pulled in tension "as-is", or in their came configuration rather than as a machined tensile test specimen. This provided a real-world comparison between samples as cracks present in the old cames were not eliminated by machining. The results of this testing are summarized below. Note that the sample designations here are specific to this testing and do not refer to Table I sample designations.

Tensile Test Sample	Age	Ultimate Tensile Strength, psi
$\mathcal{A}$	~ 1913	1,349
$\mathscr{B}$	new	3,587
C	~ 1930	1,787
Ð	new	4,492

Table IITensile Test Results

The new cames tested were chosen based upon size to compare with older cames. This means that one new Sample,  $\mathcal{B}$ , was the same size and configuration came as Sample  $\mathcal{A}$ ; and Sample  $\mathcal{D}$  was the same size and configuration as Sample  $\mathcal{C}$ . This is shown in Figure 22. These results indicate that the strength of a new came is a minimum of two and a half times that of an old came. In other words, using a new came provides 250% more tensile strength than the old cames. Since the lead cames are the structural framework for the glass, this translates into a much greater ability to withstand the weight of the glass and the winds loads to which windows are subjected. This is significant because SEM examination of an older crack fracture surface showed a fracture mode consistent with an overload failure, the same type of failure a tensile test produces.

The reportedly common practice of allowing a buckled window to settle and pressing it flat again will not heal the cracks that were instrumental in producing the lowered tensile strength of the two older cames. In fact, attempting to press buckled and distorted came walls back into position can extend cracks already present, as well as cause new ones, when the stretched metal is forced to lie flat again. This is a simple geometric response. The came walls cannot "unstretch".

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#### **Chemical Analysis**

Chemical analysis was performed on came Samples A, B, and C to determine if any compositional differences existed between the older leads from the early 20<sup>th</sup> century and new lead ordered to "restoration quality". The results are summarized below.

Element	Composition, weight %			
	Sample A, 1913	Sample B, 1930	Sample C, new	
Antimony	0.12	0.14	0.78	
Arsenic	< 0.0002	< 0.0002	0.001	
Bismuth	0.080	0.025	0.018	
Calcium	< 0.0002	< 0.0002	< 0.0002	
Copper	0.004	0.033	0.027	
Iron	< 0.0002	< 0.0002	< 0.0002	
Lead	Remainder	Remainder	Remainder	
Lithium	< 0.0002	< 0.0002	< 0.0002	
Nickel	< 0.0002	< 0.0002	< 0.0002	
Silver	0.004	0.005	0.006	
Sulfur	< 0.001	< 0.001	< 0.001	
Tellurium	< 0.0002	< 0.0002	0.0002	
Tin	0.031	0.064	0.26	
Zinc	0.0004	0.0003	0.0005	

### Table IIIChemical Analysis Results

These analysis results indicate that the lead cames from ~ 1913 and 1930 (Samples A and B, respectively) are very similar and are similar to two Unified Numbering System alloys: L52505 Lead-Antimony alloy and L52510 99.8% Lead. This is consistent with manufacturing efforts of this time to produce high purity lead for window cames.

The new "restoration lead" (Sample C) contains a much higher level of antimony and tin than the older lead. This alloy is similar to many UNS alloys, among them: L52560 Bullet Alloy, L52615 Lead-Base Die Casting Alloy, etc.

The new lead contains a larger amount of elements known to produce something known as solid-solution hardening effects (i.e., antimony, bismuth, arsenic, tin, etc.). This means that lead with the chemical composition of the new lead would be slightly stronger than lead with the chemical composition of the old lead, even if both samples were in a new, uncracked condition. A stronger alloy is capable of withstanding service conditions better than a weaker alloy.

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#### HISTORY AND DISCUSSION

Post Industrial Revolution lead is a thoroughly modern substance, with a purity and control of composition that medieval glass window artisans could not begin to imagine. In fact, at the time that many of the famous European windows were created, the producers of their leaden cames did not possess the ability to determine what other metals were alloyed with the lead, let alone refine the lead to modern standards or produce consistent alloys. Analysis of medieval came has indicated that the lead of this time contained silver, antimony, copper, tin, etc., in varying amounts.

By the mid 19<sup>th</sup> century, modern refining processes were developed that enabled the extraction of these extraneous metals from the lead. This much purer came was then thought to be superior to the older, alloyed variety and was used extensively throughout the heyday of leaded window production in the United States.

Unfortunately, removal of the alloying elements resulted in a much weaker came. The unrefined medieval lead was much better at handling the loading imparted by the glass it contained and the wind forces to which it was exposed. Modern "restoration quality" lead is reported to be based upon analysis of some medieval cames. As the chemical analysis performed in this investigation shows, "restoration" lead contains a higher percentage of elements known to produce solid-solution hardening of lead than the older, late 19<sup>th</sup> and early 20<sup>th</sup> century lead. While this means that the restoration lead is stronger than the older lead of higher purity, even this lead, and its medieval counterpart, will eventually fail in service.

The reason the lead will eventually fail in service is due to the nature of the substance. Lead is unresponsive to heat treatment and can spontaneously recrystallize at room temperature, making work-hardening for any useful period of time impossible. Due to its low melting temperature, lead is subject to creep at the temperatures in which it is normally used. Creep is a slow, plastic (i.e. permanent, doesn't return to its shape once stress is removed) deformation of materials under constant stress, like a window came supporting glass. This means that the buckling and came cracking exhibited by many aging 19<sup>th</sup> and 20<sup>th</sup> century windows is an inherent and unavoidable structural failure of the lead came resulting from the combination of modern refining processes and the nature of lead itself. While medieval and restoration lead will be better at handling service stress due to their different chemical makeup, even these leads will eventually fail in a similar manner.

Since came cracking cannot be effectively mended without producing a low-quality resoldered joint, and pressing a window flat again can lead to glass damage and came cracking, releading presents the best structural solution to the problem of a buckled window.

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The aesthetic effects achieved by the synergy of a particular lead came profile and stained glass have led to comparison with antique furniture. However, a failed structural support system is in no way comparable to the cosmetic scratches, dents, or finish cracking of desirable antiques. No reputable antique furniture dealer would suggest repairing a splintered, sagging bureau leg with plywood braces and some nails to "preserve the history" of such structural damage. Yet this is effectively the method advocated by proponents of the press and solder technique. Releading a window does not alter the artwork of the glass and its arrangement. It merely replaces failed framework, allowing the artwork it supports to be enjoyed by another generation. A reputable restorer will seek to preserve the aesthetic effects of the lead/glass interplay by utilizing appropriate replacement caming to preserve those effects.

Glass is a brittle substance and breaks easily. Once a window has buckled, its glass panes are subjected to loads never intended by the original window designer. The cracked and stretched cames can no longer bear the loads they originally did and these loads are then transferred to the glass panes. This is a recipe for the destruction of the glass. In an effort to preserve "authentic" lead, a window owner or repair facility using the press flat technique sets up a situation where the likelihood of damage to glass is greatly increased. This appears to be a classic throw-the-baby-out-with-the-bathwater situation. Releading provides the opportunity to preserve the artwork in a stained glass window by removing the loads from the glass and allowing us to view the artistry of that window as originally intended: flat, structurally sound, and with the original glass preserved from breakage by buckling forces. As tensile testing indicates, lead is a weak, low-strength material. Buckling of the frames and associated overload cracking of the came walls is typical of structural failure of a load-bearing member. Given traditional came profiles, this appears to be inevitable and a window owner will eventually have to decide whether to save and preserve the old lead, or to save and preserve the glass panes.

#### CONCLUSIONS

Several conclusions can be drawn from the analysis results and review of repair and releading techniques. These are presented below as a bullet list. For greater detail, refer back to History and Discussion section, as well as individual testing results sections.

- Modern refining techniques produced lead of much greater purity for use in mid-19<sup>th</sup> century to mid 20<sup>th</sup> century windows. This lead is very different from medieval lead and its modern Restoration lead counterpart.
- The lead of greater purity is a weaker metal than the alloyed medieval lead and modern Restoration lead. As a result, it is less able to withstand glass weight and wind loads than its alloyed relatives. Came wall stretching and cracking will eventually result.

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- Pressing flat a buckled window does not repair cracks in the cames. The pressing process is likely to propagate existing cracks and create new ones.
- Resoldering old joints in cames results in poor joint quality and can induce further cracking at the solder pool toe. This does not restore the window lead framework to "good as new" condition.
- Window buckling due to lead framework structural failure transfers loads to glass panes that were previously handled by the lead. This is a recipe for glass breakage due to its inherent brittleness.
- Modern "restoration quality" lead came consists of an alloy based upon chemical analysis of some medieval leads. Use of this alloyed lead in restoration of windows should result in the greater ability of the restored lead framework to withstand service loads over the purer lead used in the late 19<sup>th</sup> and early 20<sup>th</sup> centuries. However, as with all structural frameworks, even the restoration lead will eventually require replacement.
- The cracks in cames visible to the naked eye are not the only cracks present. Soldering over visible cracks does not eliminate these microscopic cracks. Cracking weakens the cames and reduces their ability to withstand service loads.
- Tensile testing revealed new came strength to be at minimum 250% higher than cracked came strength.
- Came cracking is an inevitable result of service due to the inherent ability of lead to creep at normal use temperatures and to resist heat treatment and work hardening procedures used regularly with other alloys. While the solid-solution strengthening possible with certain alloying conditions makes stronger cames available, even these will eventually experience structural failure due to the behavior of their lead base.



Figure 1: Cracks in Sample A as-received, arrows.

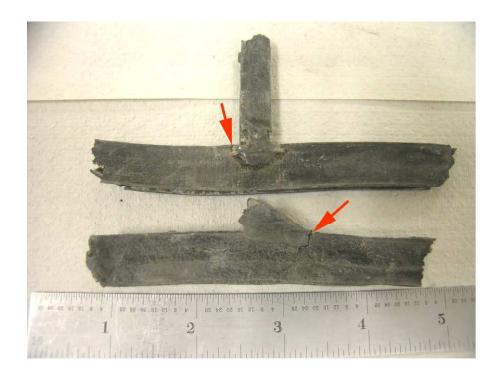


Figure 2: Cracks in Sample A joints, as-received, arrows.



Figure 3: Close-up of crack in top joint of Figure 2, arrow. Note also joint profile and appearance.

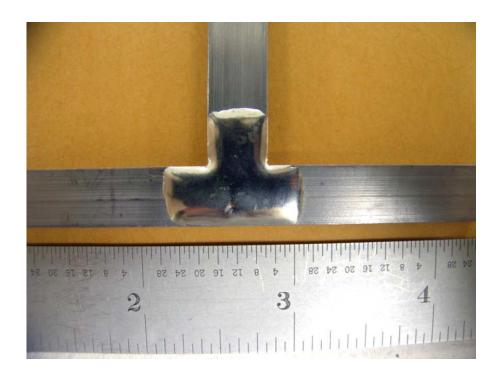


Figure 4: New joint.



Figure 5: Fracture surface of Sample A, arrow. Mag. 12X



Figure 6: Fracture surface of Sample B, arrow. Mag. 12X

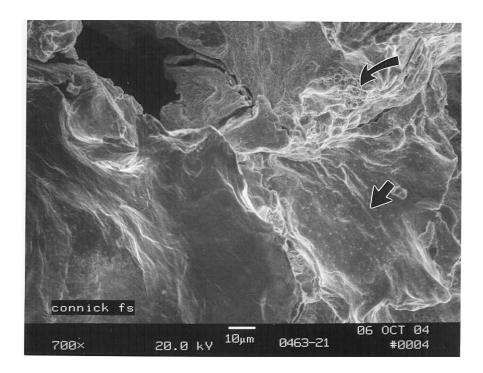


Figure 7: Sample A, ~ 1913, fracture in SEM showing ductile dimple rupture, curved arrow, and wide patches of ductile tearing, straight arrow. Mag. 700X

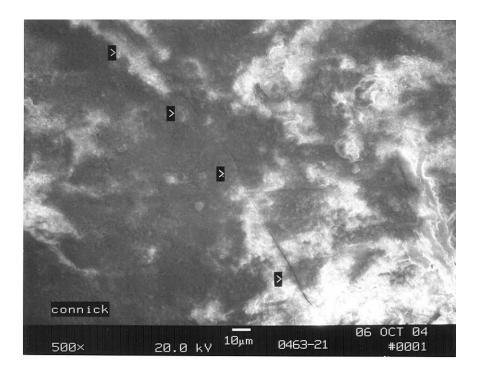


Figure 8: Cracking on flat surface of Sample A, arrows, proceeds into lead beyond oxide. Mag. 500X

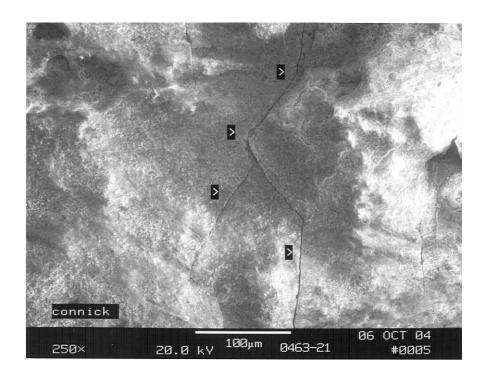


Figure 9: Cracking on flat surface of Sample A, arrows, proceeds into lead beyond oxide. Mag. 250X

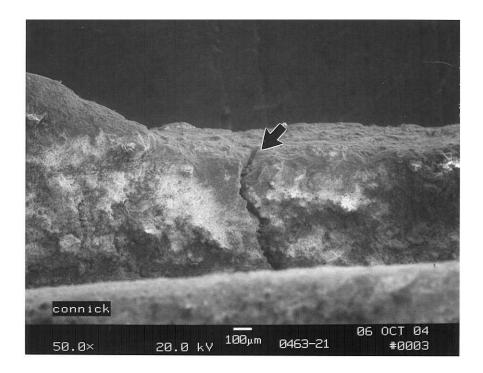


Figure 10: Crack in came near solder, Sample A, arrow. Mag. 50X

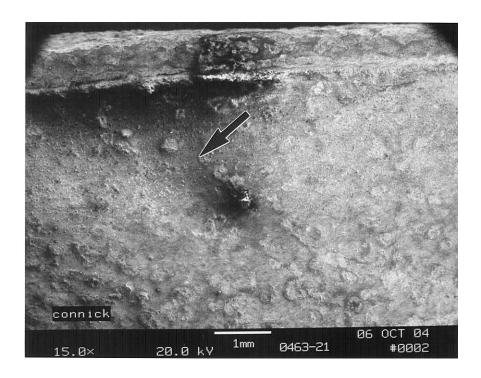


Figure 11: Location of cracks on Sample A came, arrow. Mag. 15X

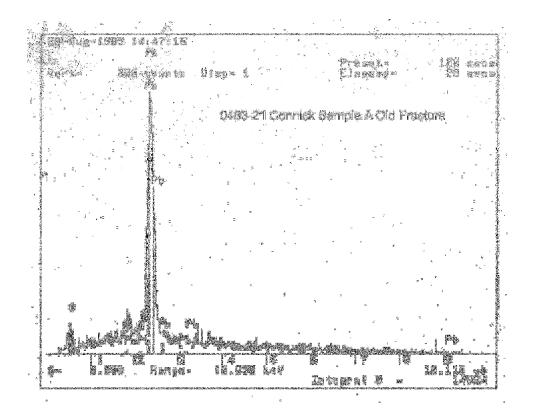


Figure 12: Sample A old fracture EDS spectrogram.

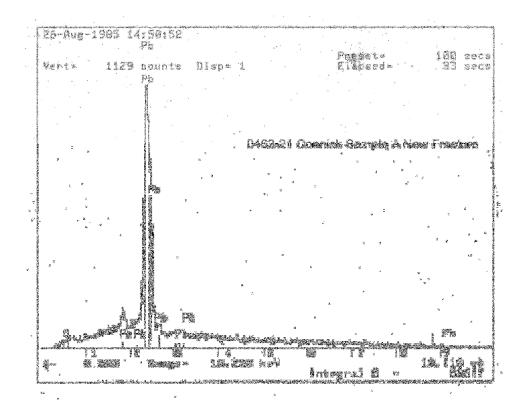


Figure 13: Sample A new fracture EDS spectrogram.

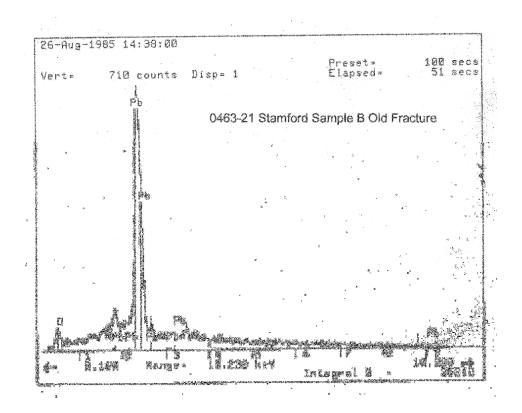


Figure 14: Sample B old fracture EDS spectrogram.

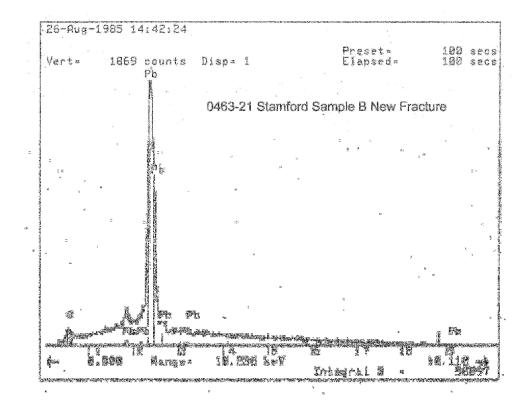


Figure 15: Sample B new fracture EDS spectrogram.

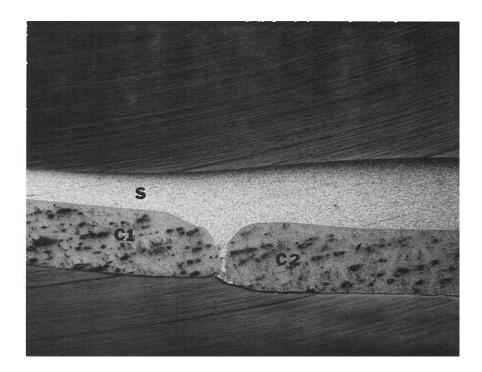


Figure 16: New solder (S) joint joining two cames (C1 and C2). As-polished. Mag. 25X

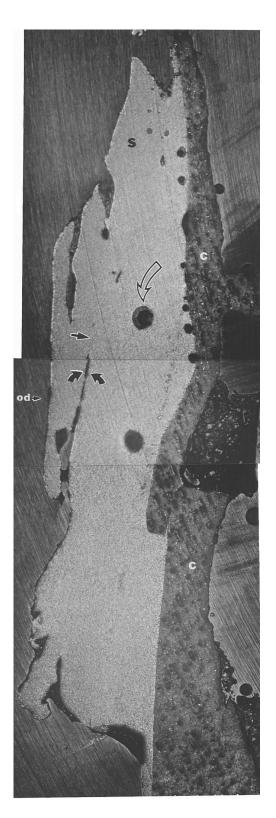


Figure 17: Resoldered joint showing solder(s) containing porosity (curved clear arrow) and lack of fusion (between curved solid arrows). Note that the lack of fusion extends to the small straight arrow above the two curved solid arrows. Original Magnification: 18.75X

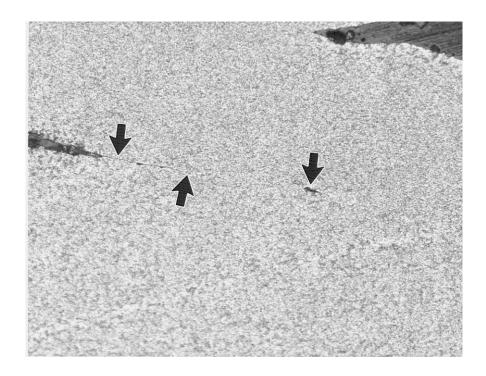


Figure 18: Higher magnification view of lack of fusion in Figure 17, arrows. As-polished. Mag. 100X

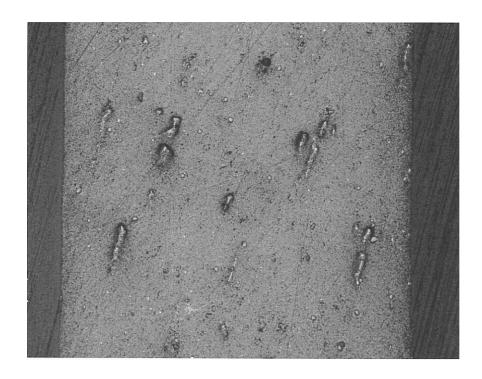


Figure 19: New came in cross-section. As-polished. Mag. 120X

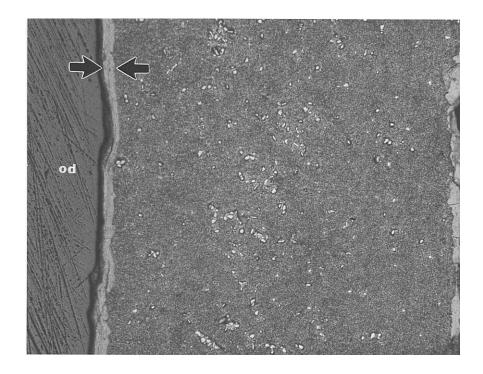


Figure 20: Oxide layer on OD of Sample A, arrows. As-polished. Mag. 150X

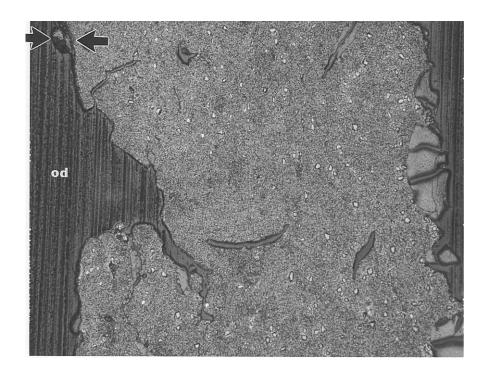


Figure 21: Oxide layer remnant on OD of Sample B, arrow. As-polished. Mag. 150X



Figure 22: Cames for tensile testing.

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