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Date: **05/09/2017**

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A Thesis Presented to

The Faculty of the Advances Education in General Dentistry, Two-Year Program

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In Partial Fulfillment of the Requirements for a

Degree of Masters of Science in Oral Biology

By

Alexandre Vo, Captain

Royal Canadian Dental Corps

January 2016

shear bond strength of a new bioactive luting cement on a CAD/CAM high-density hybrid composite material

A REPORT ON

A project to investigate the bonding strength of a dental luting cement on a high-density composite materials for CAD/CAM technology.

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shear bond strength of a new bioactive luting cement on a CAD/CAM high-density hybrid composite material

ABSTRACT

Background. None of the current commercially available cements meet the all of the desired criterion of luting agents. **Purpose.** This investigation evaluated the shear bond strength of a hybrid luting cement made of calcium-aluminate/glass ionomer (CA/GI) applied to a composite CAD/CAM block. **Materials and Methods.** CA/GI luting cement (Ceramir C&B) specimens mounted on Cerasmart composite blocks were tested using a universal testing machine after incubation at 37 C in PBS solution for 24-hour and 7-day, and compared to a self-adhesive resin cement (RelyX Unicem, control). **Results.** Ceramir C&B has a significantly lower shear bonding strength than RelyX Unicem when applied to a CAD/CAM composite material. However, no significant statistical difference in shear bond strength was found within Ceramir nor RelyX Unicem subgroups at 24-hour and 7-day intervals. **Conclusion.** Hybrid of calcium-aluminate/glass ionomer may not be the cement of choice for luting high-density composite restorations when preparation design cannot provide predictable retention and resistance to dislodgement.

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BACKGROUND

Over the years, many luting cements have been developed to fill in the void between an indirect restoration and the tooth in order to prevent dislodgment of the restoration during function (Hill 2011). This viscous material placed between the tooth structure and the restoration, hardens to bind the two components together primarily through micromechanical interlocking. Luting cements are also designed to fill the microscopic gaps between the two luted surfaces to prevent leakage, and increase support and retention of the prosthesis. There are two main categories of luting cements, based upon their setting reaction: acid-base reaction and polymerization. None of the currently available cements have shown to be ideal in terms of strength, biocompatibility, working and setting time, water sorption, solubility and microleakage.

Zinc phosphate

Among the acid-base reacting cements, zinc phosphate is known for its long record of success. For that reason, it is being used as a reference when comparing luting cements. It has a high compressive strength, and its stiffness makes it suitable for long-span or cantilevered fixed partial prosthesis (Hill 2011). Its high early strength makes it fit for luting cast metal post-core (Habib 2005).

However, like any ionic agent used intra-orally, it is susceptible to acid erosion (Stannard 1989). Early on, zinc phosphate cement is sensitive to moisture and may erode (Curtis 1993). The low pH of the phosphoric acid zinc phosphate cement contains is known to cause sensitivity when it is applied on thin dentin walls (Phillips 1991, Pameijer 1984). Such clinical situation may warrant placing a varnish on the dentin prior to cementation in order to prevent such irritation, undermining the retention provided by the luting agent (Felton 1987).

Polycarboxylate

Polycarboxylate cement was reported to be less irritating to the pulp tissue due to larger molecular weight of the polycarboxylic acid. It also has some chemical adhesion to tooth structure. It is mostly used on metal and metal-ceramic restorations (Hill 2011).

Its short working time makes its use challenging when cementing multiple restorations at once. Its higher viscosity makes seating of the restoration less reliable. For that reason, modifying the powder-liquid ratio may improve workability of the cement but will, on the other hand, negatively affect its physical properties (Bruce 1989) and solubility (Osborne 1991) due to the increased amount of acid. Polycarboxylate cement has the lowest compressive strength of luting cements (Mesu 1983). Its high degree of plastic deformation limits its use to single-unit restorations (Oilo 1978). Just like zinc phosphate cement, it is prone to dissolution in the oral environment (Stannard 1989). It is nowadays most often used as long-term provisional luting agent for non-metal restorations. (Hill 2011).

Glass ionomer

Glass ionomer cement (GIC) was introduced as an improvement in physical properties and pulp response from silicate cements (Primus 2013). It adheres to both tooth structure and base metal, which explains why it is indicated for luting metal and metal-ceramic restorations (Hill 2011). Another advantage of GIC cement is its fluoride release and uptake (Thornton 1986), which helps reduce demineralization at the margins of the restoration (Stannard 1989, Tsanidis 1992). Clinical studies have supported the claim that this fluoride release is significant in preventing carious lesions (Tyas 1991, Svanberg 1992).

The low modulus of elasticity of GIC limits its use to single-unit restorations, or short-span fixed partial prosthesis exempt from significant occlusal load (Hill 2011). It is also susceptible to moisture contamination during the setting phase (Um 1992), as water absorption and loss of cations ultimately leads to erosion of the cement (MacLean 1988). Glass ionomer should neither be allowed to desiccate during maturation to prevent crazing (Mathis 1989). When thin dentin walls remain, pulpal irritation is a concern when using GIC due to its lower pH and cytotoxicity of some of its components (Smith 1986, Bapna 1994). Therefore, sensitivity has been reported with this material as well. GIC demonstrates higher creep values (Cattani-Lorente 1993), which can affect clinical performance as it undergoes deformation over time (Rosenstiel 1989).

Resin-modified glass ionomer

In order to overcome the problems encountered with GIC, a small amount of monomer was added, along with initiators to allow the polymerization of the monomers to occur. The resulting resin-modified glass ionomer cement (RMGIC) led to improved physical properties, reduced sensitivity to moisture and dessication, while preserving the benefits of the conventional GIC (Sidhu 2010, Mathis 1989). However, water sorption of the resin phase and subsequent hygroscopic expansion (Knobloch 2000) has shown to decrease physical properties over time (Cattani-Lorente 1999, Nicholson 2008). Cases of low-strength all-ceramic crown fractures using RMGIC have been attributed to the cement expansion (Leevailoj 1998). It is mainly indicated as a luting agent for metal and metal-ceramic restorations, as well as for alumina and zirconium ceramic crowns (Ernst 2005).

RMGIC has shown similar creep values as conventional GIC (Yamazaki 2006), and margins tend to deteriorate over time (Sidhu 2010). Just like GIC, RMGI cement margins need to be kept dry during cementation to prevent early dissolution. Finally, the resin phase makes removal of excess difficult once the cement has set. Its hydrophilic monomers have also shown to be cytotoxic to pulp tissue (Bouillaguet 1998). The toxicity of monomers in RMGIC will be discussed further with adhesive resin cements, of which they both share.

Adhesive resin cement

With increased popularity of all-ceramic restorations thanks to enhanced aesthetics, adhesive resin luting cements have become the cement of choice as they have shown to effectively bond to both ceramic and tooth structure, therefore providing better retention than conventional cements (Michelini 1995) and favorable esthetic results (Hill 2011). Adhesive resin cements have also shown to increase the strength of all-ceramic restorations and improve their fracture resistance (Burke 1994, 1995, 2002). They are also indicated for metal copings that have tooth preparations with compromised retention and resistance form (Pegoraro 2007). In contrast with acid-base luting cements, adhesive resin cements have shown less solubility and microleakage (White 1994).

One drawback of resin cements, due to the application protocol, is moisture control is critical. Once the resin cement has set, removal of excess is of the utmost importance and could pose a problem where access is limited (Hill 2011). Additionally, toxic substances may elute from the composite resin, since the resin cement does not completely polymerize (Alshali 2013): the concentration of eluted substances is inversely related to the degree of conversion of the composite resin (Durner 2012). The unreacted monomer resin can leach out and irritate the pulp tissue (Goldberg 2008). Composite resin eluates may cause cytotoxic and allergic reactions, and genome mutations in the host cells (Schmalz 2009). Some controversy persists whether composite resin has estrogen-like effects: even at very high dose, levels remained very low compared with estradiol (Hashimoto 2000), but clinical significance remains to be clarified (Wada 2004). Monomers found in composite resin have shown to promote bacterial growth and aggregation, thus plaque formation (Hansel 1998, Schmalz 2009) Moreover, water sorption by the organic matrix as stated above causes expansion and decreases physical properties of the resin cement, but less than RMGIC (Mese 2008, Musanje 2001). Over time, nanoleakage within the hybrid layer has been observed, compromising the bond at the dentin-resin interface (Sano 1995).

To sum up, the ideal luting cement should have a low film thickness, long working and short setting time. Once set, it should display high compressive and tensile strength, a modulus of elasticity similar to dentin. It should be minimally irritating to the pulp tissue and biocompatible. It should be hydrophilic in order to minimize water sorption and solubility, thus microleakage. An ideal luting cement should also prevent development of plaque. None of the current commercially available cements meet the all of the desired criterion of luting agents, hence the need to pursue further research for a truly universal luting cement.

Calcium-aluminate/glass ionomer

Calcium-aluminate (CA) cement is part of the group of chemically-bonded ceramics (CBC). These bioceramics are based on calcium phosphate salts which can be made to cure *in vivo* and represent an interesting replacement material for the natural calcium phosphate found in mineralized tissues such as teeth and bones. This property makes it bioactive. By definition, a bioactive material elicits a specific response at the interface of the material which results in the formation of a bond between the tissues and the material. (Cao, 1996). CA's hydrophilicity and water uptake properties are interesting features for use as dental materials: the body fluids promote the setting reaction of calcium-aluminate cement as phosphate ions are used to form apatite. As CA sets and hardens, a tight bond forms with the tooth and no gap is seen because of the precipitation of apatite towards the tooth surface that produces a predictable seal (Engqvist 2004). This is made possible in part throughout the hydration process where the pH is maintained between 8 and 11. (Hermansson 2006, Engqvist 2006). The fully hardened material maintains a basic pH throughout its service. This basic pH is the most important prerequisite for the material to be bioactive because it creates apatite on its surface when it comes in contact with phosphate-containing solutions (Lööf 2008). Additionally, the material produces an excess of calcium ions, which also contributes to its bioactivity.

General characteristics of CA cements are rapid-hardening properties, high initial strength, and excellent acid-corrosion resistance. The hardness increases with time due to a continuous hydration. As hydration progresses, the overall porosity decreases. Most of the hydration is completed after a week, but it can extend over a few months. Calcium aluminates also have good wear resistance, comparable to that of composites (Kraft 2002). As a dental filling material, it has also been shown that CA exhibits equal or greater hardness than amalgam and composite. It also shows a linear expansion of 0.2% (Kraft 2008), as opposed to a contraction for amalgam and composite. A shear bond strength in the range of 10 to 25 MPa has been reported at the tooth-CA interface, a compressive strength of 150 MPa, Young's modulus of 15 GPa and flexural strength of 50 MPa. (Kraft 2002, Lööf 2008, Lööf et al. 2004, 2005, Hermansson et al. 2008). In a complementary cytotoxicity test using the pulp derived cell response, the experimental calcium aluminate material showed no sign of toxicity, therefore safe to use on human subjects (Schmalz 2002).

Out of these findings, a new bioactive luting cement has recently made its way to the dental community. Ceramir C&B is a water-based hybrid composition comprising of calcium aluminate and glass-ionomer (GI) components that is mixed with distilled water in the form of single-use, mixing capsule. The acid-base reaction of the glass ionomer component makes the pH slightly acidic initially: the pH is around 4. Within 1 hour, the pH is neutral. and after 3 to 4 hours it reaches a basic pH of 8.5. (Jefferies 2009, Pameijer 2011). The compressive strength, film thickness, and setting time all conformed to the International Standard Organization (ISO) standard for water-based luting cements. The setting time of Ceramir is 5 minutes, while its film thickness is 15 microns. Its compressive strength is 160 MPa, which is comparable to RelyX Unicem (3M, Minneapolis, MN, USA), a popular resin cement (Jefferies 2008). Ceramir demonstrated significantly less leakage than Ketac Cem (3M, Minneapolis, MN, USA), a conventional glass-ionomer cement (Pameijer 2008). The incorporation of CA fixes the GI structure and hinders the GI from continuously leaking over time. (Pameijer 2011).

Cerasmart is a high-density composite resin material containing 65% of small and uniformly distributed filler particles of alumina- and barium-silicate (Lauvahutanon 2014). It is part of a new generation of polymer-based CAD/CAM materials that has a dispersed filler microstructure and is polymerized at high temperature. This process significantly increases the degree of conversion of the polymer and thus improves physical properties of the material (Mainjot 2016) and resistance to wear (Lauvahutanon 2015). Flexural strength, Weibull modulus, hardness and density all improve under high pressure/high temperature (HP/HT) processing (Nguyen et al, 2012). This is even more true for filled than unfilled resin, suggesting a significant effect on the filler-matrix interaction (Phan et al. 2014). HT dispersed-filler composites have shown to be more resilient compared with glass-ceramics: they can withstand higher loads by undergoing more elastic deformation (Awada 2015). CAD/CAM composites have shown better fatigue resistance and withheld better loads - superior than masticatory forces - than their ceramic counterparts in posterior restorations, whether as crowns or occlusal veneers (Kassem 2012, Magne 2010). It has also been suggested that the incorporation of an initiator could be eliminated, thus preventing discoloration of the composite over time, but remains to be validated (Ruse 2014). Less marginal chipping and smoother margins are observed with composite materials during milling than with glass-ceramics due to reduced brittleness (Tsitrou 2007, Awada 2015). Therefore, they can be milled at very low thickness (Awada 2015, Dirxen 2013). These composite materials also display lower hardness values, causing less wear to and extending the life span of the milling burs, as compared to ceramic materials (Lebon 2015). Repairs are easier to make on CAD/CAM composite restorations and the repair material – again a composite resin - blends in nicely for favorable esthetic outcomes (Rocca 2010). However, as we would expect from any polymeric materials, physical properties appear to be negatively influenced by the water sorption. A decreased in flexural strength, in flexural modulus and in hardness has been observed. Their use is therefore recommended for single-unit restorations in the premolar area (Lauvahutanon 2014).

Although the manufacturer also recommends cementing Cerasmart with an adhesive cement, there is no mention or contraindication about its use with other luting, non-adhesive cements.

The current study served two purposes. First, it attempts to determine if there is a difference in the shear bond strength between a hybrid of calcium-aluminate/glass ionomer bioactive luting cement and a popular resin cement. The null hypothesis is that there is no statistically significant difference in shear bond strength between the two materials (Ceramir and Unicem). The alternate hypothesis is that there is a statistically significant difference in shear bond strength between the two materials.

Second, we tried to determine if there is a difference in the shear bond strength at 24-hour and 7-day mark for both cement tested. The null hypothesis is that there is no statistically significant difference in the shear bond strength at the 24-hour and 7-day mark. The alternate hypothesis is that there is a statistically significant difference in shear bond strength between the 24-hour and 7-day intervals.

MATERIALS AND METHODS

A total of 80 specimens consisting of GC Cerasmart CAD/CAM blocks size-12, A2-HT (GC, Tokyo, Japan, lot 1503091) were divided in four groups of 20 samples as follow: two of those groups were assigned to the experimental cement, Ceramir C&B (Doxa Dental, Uppsala, Sweden, lot 1533388), while the remaining two were assigned to the control cement, RelyX Unicem (MaxiCap, A2 shade, 3M ESPE, Minneapolis, MN, USA, lot 592742). For each cement, one group was tested 24 hours after bonding, while the other was tested 7 days after bonding (Table 1).

Substrate	Cerasmart CAD/CAM Blocks (80)			
Cement	Ceramir C&B (40)		RelyX Unicem (40)	
Groups	C1: 24-hour (20)	C7: 7-day (20)	U1: 24-hour (20)	U7: 7-day (20)

Table 1: Experimental and control groups observed over two different time intervals. Numbers of samples per group shown in parentheses.

Samples were fabricated following the ISO 29022 standard for testing shear bond strength. Samples were made from sectioning of Cerasmart blocks using a diamond wafering blade (Series 15HC No. 11-4245, Buehler, Lake Bluff, IL, USA) mounted on a Linear Precision Saw (Isomet 5000, Buehler, Lake Bluff, IL, USA) (Figure 1).

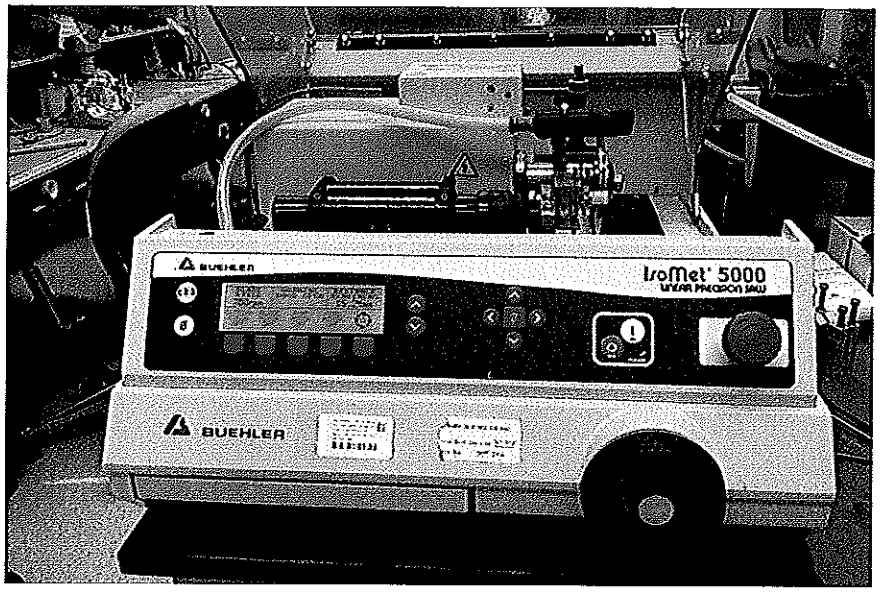

Figure 1: Linear Precision Saw

Before initiating the bonding procedure, the samples were embedded in orthodontic resin (Great Lakes Orthodontics, Tonawanda, NY, USA) poured in 1.25-inch plastic ring form (Buehler, Lake Bluff, IL, USA) mounted on a glass slab coated with a separator (Great Lakes Orthodontics, Tonawanda, NY, USA), ensuring that one surface of the substrate remains uncovered for bonding procedures (Figure 2). Samples were then polished using an abrasive paper (CarbiMet S 8 inches 240 grit, Lake Bluff, IL, USA) mounted on a polisher (Ecomet 6 Variable Speed Grinder-Polisher and Automet 3 Power Head, Buehler, Lake Bluff, IL, USA) in order to obtain a standardized surface corresponding to the grit (60-70 microns) of a CEREC 12 S bur (Sirona USA, Charlotte, NC, USA) (Figure 3).

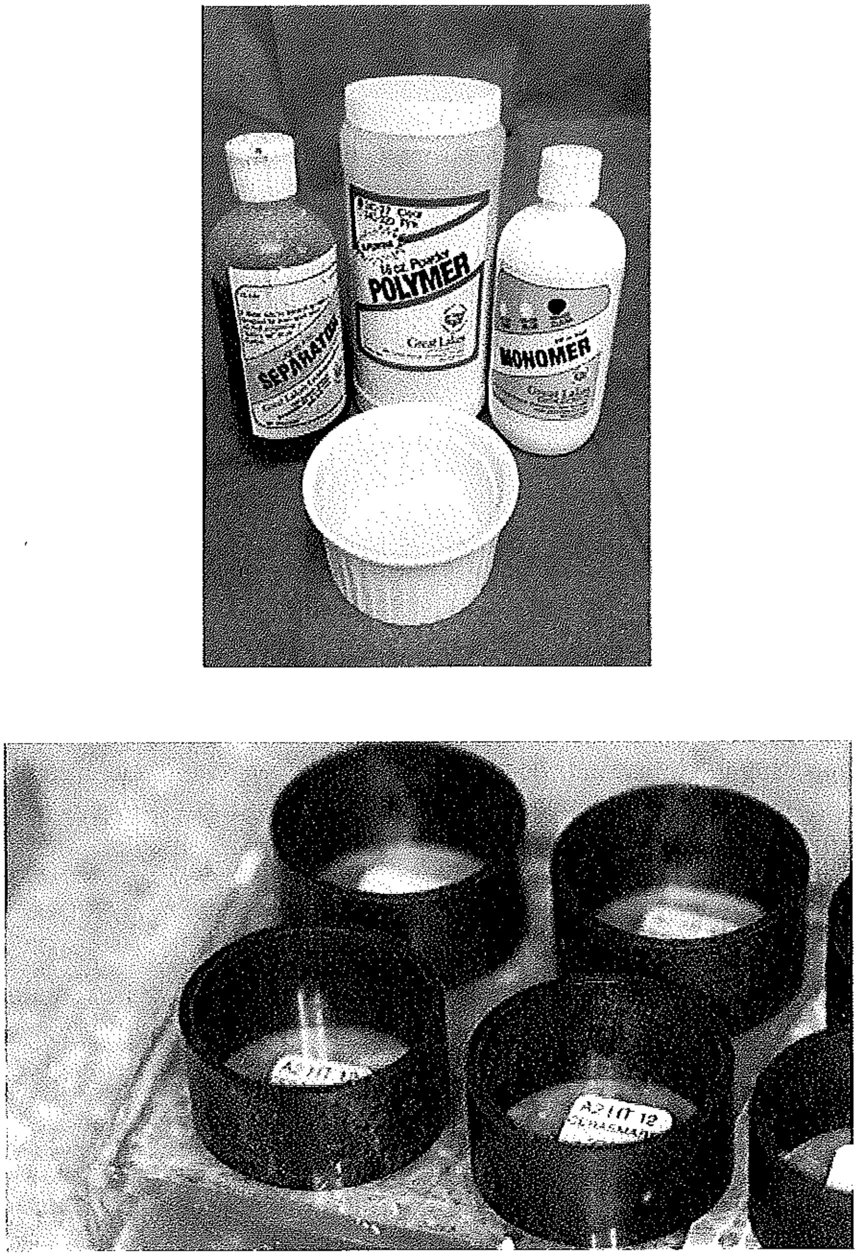

Figure 2. Separator and orthodontic resin (above). Samples of Cerasmart blocks in plastic ring forms mounted on lubricated glass slab (below)

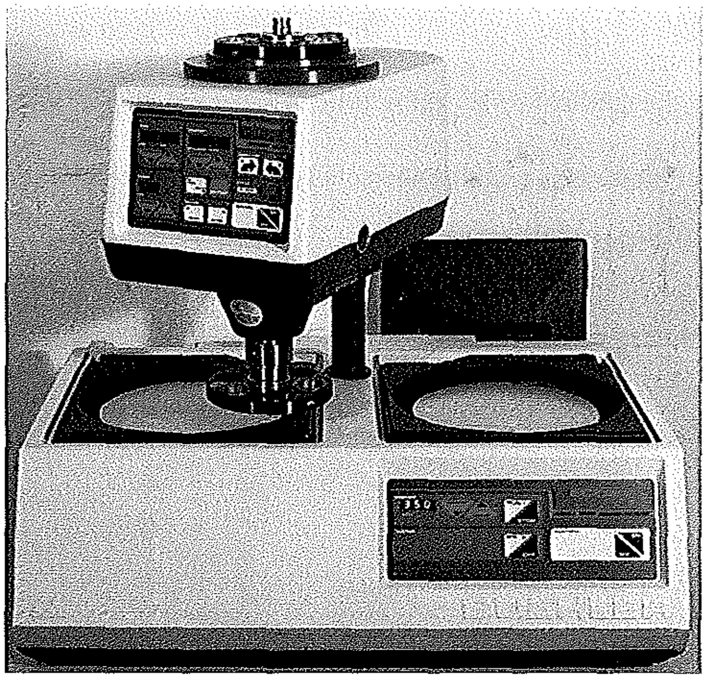

Figure 3. Polisher, using 240-grit abrasive paper

The substrate was then preconditioned according to the manufacturer's instructions. As per the manufacturer's guidelines, Ceramir C&B did not require a surface treatment of the substrate. As for the substrate itself - the nano-ceramic hybrid composite Cerasmart - no indication has been provided for the use of Ceramir C&B. Therefore, no surface treatment of the Cerasmart blocks was performed when used in the Ceramir groups. In the control group using RelyX Unicem, the substrate requires sand blasting with 50-micron aluminum oxide particles (Renfert, Hilzingen, Germany) then wiped with alcohol and dried.

The samples were inserted into the bonding clamp containing a white plastic button mould with a hole diameter of 2.38 mm (Ultradent, South Jordan, UT, USA) (Figure 4). The mould is then half-filled with the cement and the latter is cured according to the manufacturer's instructions. The Ceramir C&B capsule is put in the activator, the tip away from the handle, which is pushed and held down for 5 seconds. The capsule is then triturated (Automix, Kerr, Orange, CA, USA) at 3400 CPM for 5 seconds. The capsule tip is spun around 180 degrees to release its content using the applicator. Working time is approximately 2 minutes. Self-curing Ceramir reaches its gel phase at 3 minutes and sets completely after another 5 minutes, for a total setting time of 8 minutes.

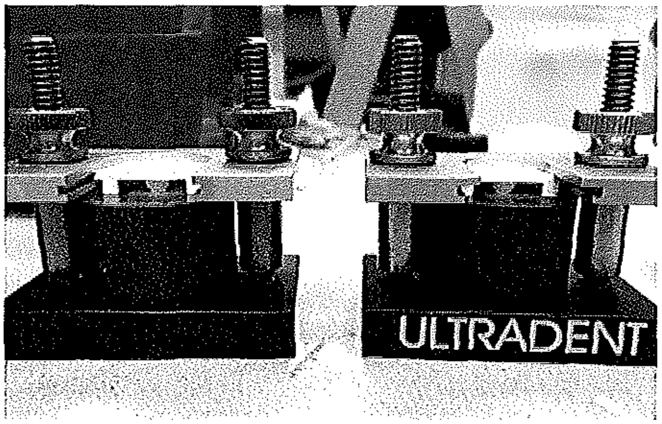

Figure 4. Bonding clamp

RelyX Unicem capsule was first inserted in the activator and the handle pressed down for 2 to 4 seconds. It was then mixed in a triturator for 15 seconds at 3400 CPM. The dual-cure cement was dispensed through the nozzle directly onto the bonding surface and was light-cured for 20 seconds (3M Paradigm, Minneapolis, MN, USA).

The samples were stored in phosphate buffered saline (Sigma-Aldrich, St. Louis, MO, USA) solution at 37 C in an incubator (Vacucenter vacuum dryer, Salvis Lab, Rotkreutz, Switzerland) (Figure 5) for 24 hours and 7 days prior to testing, based on the group. Shear bond strength was assessed on a universal testing machine (Alliance RT/5, MTS, Eden Prairie, MN, USA). Immediately after removal from water, the samples were placed into the test base clamp

(Ultradent, South Jordan, UT, USA) and aligned under the testing crosshead (0.5 mm/min) with the edge centred over the cement button and flush against the substrate (Figure 6).

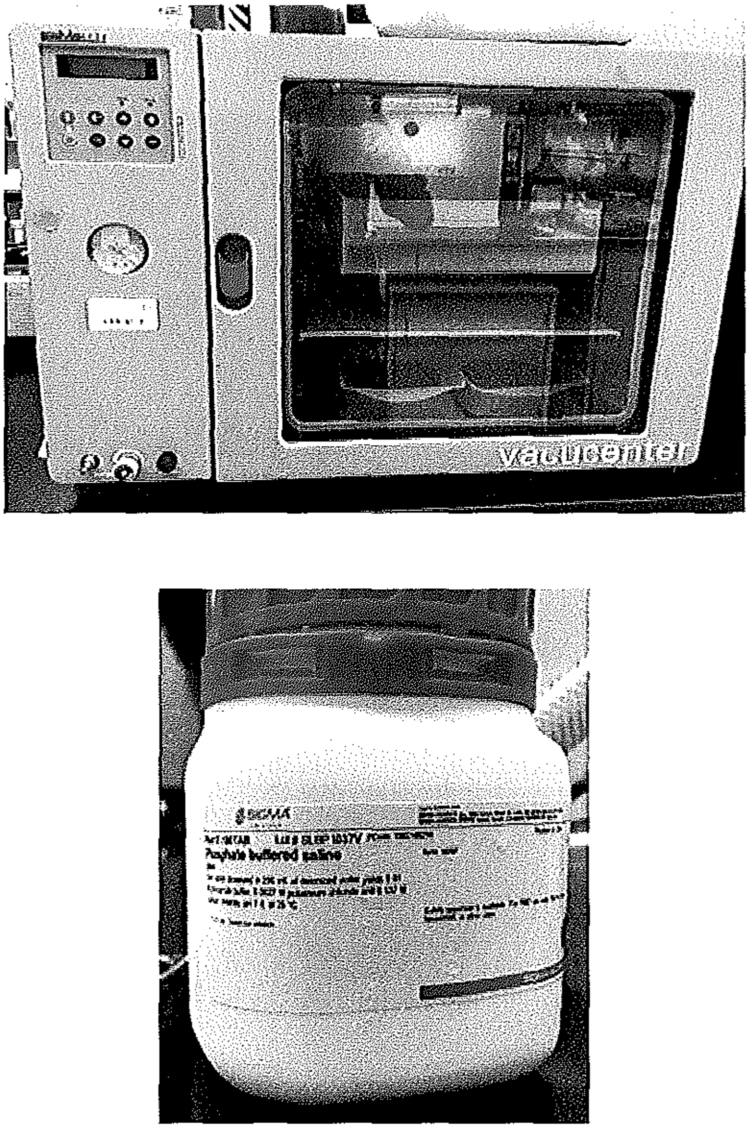

Figure 5. Incubator kept at 37 C (above). Specimens were stored for 24 hours or 7-day, based on their respective group, in phosphate buffered saline solution (below)

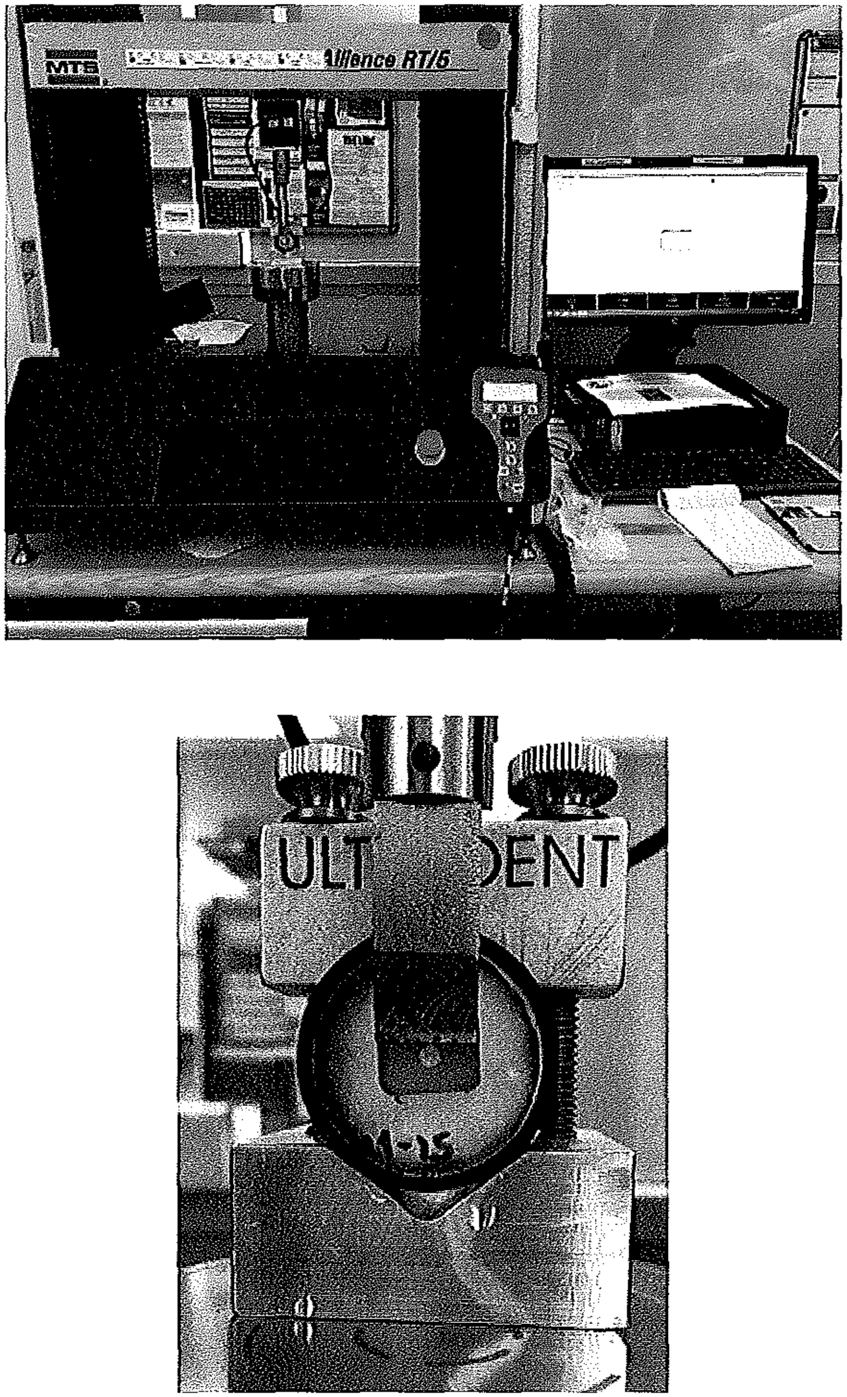

Figure 6. Universal testing machine (above). Plastic ring form with embedded sample, mounted on test base clamp, aligned with testing crosshead (below).

Measures were recorded using the MTS TestSuiteT Multipurpose Elite Software (MTS, Eden Prairie, MN, USA), as the maximum force (F) per surface area (A) prior to failure of the bond. The bond strength (σ) was then calculated using the formula $\sigma = F/A$, where

σ is the stress in MPa;

F is the force in N; and

A is the bonding area in mm^2

The bonding area is constant and is calculated as $A = \pi(2.38/2)$^2 = 4.45 mm^2 Failing samples from the Ceramir groups were observed at 10x magnification (Measurescope MM-22, Nikon, Japan) and using a measuring recorder (Quadra-Check 200, Vision Engineering, England) (Figure 7). Cohesive failures occurred within the cement, and adhesive failures occurred at the substrate/cement interface. When more than 75% of the bonding area appeared to have failed within the cement, the specimen was classified as a “cohesive” failure. The failure was defined as “adhesive” when more than 75% of the bonding area appeared to be clean substrate. When 25–75% of the failure was both adhesive and cohesive, these failures were considered “mixed”.

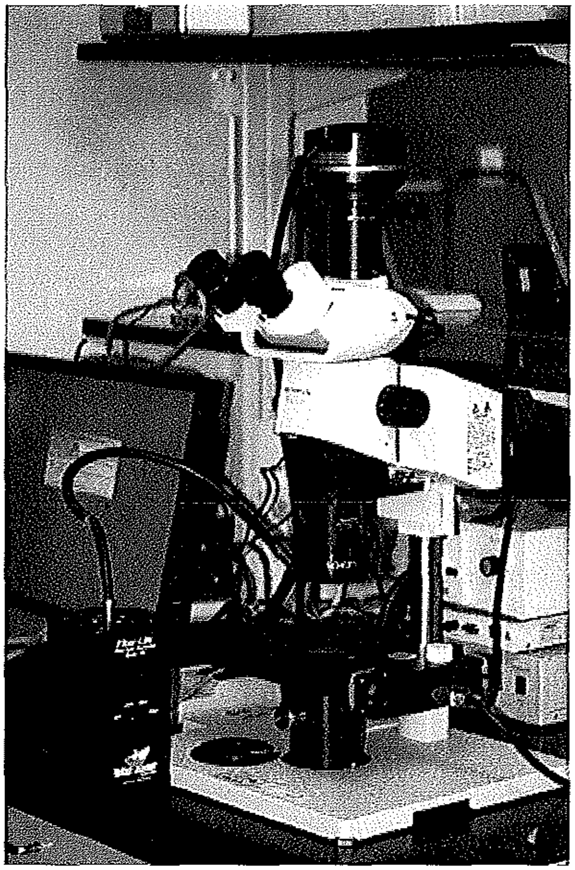

Figure 7. Measurescope MM-22

A statistical analysis was performed to test the significance of differences in the shear bond strength of the tested cement (Ceramir C&B) compared to a commonly used self-adhesive cement (RelyX Unicem), and how it performs at the 24-hour and 7-day mark.

Data analysis

The data was processed using a two factor ANOVA followed by independent sample t-tests corrected for multiple comparisons. If the data is not normally distributed with equal variance, the equivalent non-parametric test was used. A Weibull analysis was performed to assess the probability of failure versus the applied stress, hence, determining the presence of flaws in the material.

RESULTS

The current study served two purposes. First, it attempted to determine if there is a difference in the shear bond strength between a hybrid of calcium-aluminate/glass ionomer bioactive luting cement and a popular resin cement. The null hypothesis is that there is no statistically significant difference in shear bond strength between the two materials (Ceramir and Unicem). The alternate hypothesis is that there is a statistically significant difference in shear bond strength between the two materials.

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Two-way ANOVA

A two-way ANOVA test was performed in order to answer those two questions. Ceramir demonstrated a shear bond strength of 0.63 +/- 0.48 MPa and 1.14 +/- 0.59 MPa at 24-hour and 7-days respectively, while RelyX Unicem demonstrated a shear bond strength of 5.76 +/- 1.49 and 6.04 +/- 1.58 MPa (figure 8). The outcome of this statistical analysis demonstrates that there is a statistically significant difference in the shear bond strength between Ceramir, a bioactive luting cement, and RelyX Unicem, a self-adhesive resin cement. Ceramir C&B has a significantly lower shear bonding strength than RelyX Unicem when applied to a CAD/CAM composite material. However, no significant statistical difference in shear bond strength was found within

Ceramir nor RelyX Unicem subgroups at 24-hour and 7-day intervals. Therefore, we have to reject the null hypothesis to the first question. To the second question, the two-way ANOVA test validates the null hypothesis as there is no statistical significant difference between the 24-hour and 7-day intervals within one material. Refer to Appendix A for complete ANOVA analysis.

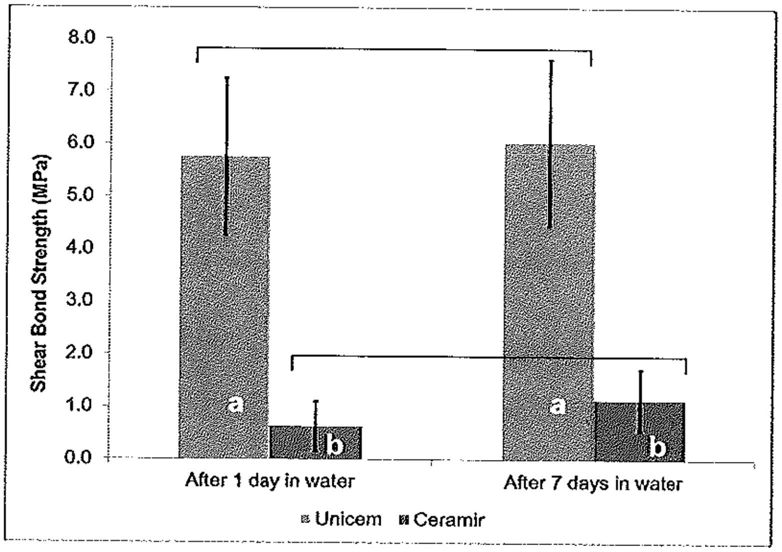

Figure 8. Shear bond strength vs. time under PBS; columns with different case letter is significantly different at that specific time frame ($p < 0.001$). The brackets on top of the columns indicate shear bond strength is not significantly affected by hydrolysis after 7 days of storage in water ($p > 0.05$).

Failure of the Ceramir specimens

Samples of the two Ceramir groups were observed under microscope to determine the type of failure that occurred under shear stress. Failure was classified as either adhesive, cohesive, or mixed. An adhesive failure was determined if less than 25% of the surface bonding area was covered by cement. A mixed failure was described as 25% to 75% of surface area with retained cement. Finally, if more than 75% of the surface area was still covered with cement, then a cohesive failure was considered.

Under 10x magnification (Measurescope MM-22, Nikon, Japan) and measuring recorder (Quadra-Check 200, Vision Engineering, England), 17 of the 20 samples in the 24-hour group were deemed to have undergone adhesive failure, compared with 16 of the 20 samples of the 7-day group. Mixed failures were observed on 3 samples of both the 24-hour and 7-day group. Only 1 cohesive failure was observed, which happened in the 7-day group. Failures for all 40 samples are shown at Appendix C.

Type of failure	24-hour	7-day
Adhesive	17	16
Cohesive	0	1
Mixed	3	3

Table 2: Type of failure for the Ceramir groups at 24-hour and 7-day intervals. Adhesion failure stands out as the main type of failure for both periods.

Weibull analysis

The Weibull analysis helps defining the probability of failure versus applied stress. As brittle materials can fail at a lower level of stress that expected because of flaws in the materials themselves, it assists in predicting and assessing the strength more sensibly. A higher Weibull modulus would translate in a more dependable material as less flaws are present within. In this case, the Weibull analysis reveals a higher Weibull modulus for RelyX Unicem than for Ceramir C&B (figure 2). Therefore, it tends to demonstrate that the tested cement, Ceramir C&B, shows more flaws in its material than our control cement, Unicem. Hence, Ceramir appears less predictable than Unicem. Complete data for Weibull analysis is sown in Appendix B.

DISCUSSION

The high ratio of adhesive failure of the Ceramir luting cement unequivocally demonstrates a weak integration or adhesion of the tested luting cement to the substrate, Cerasmart, a high-density hybrid composite material. As the ANOVA has shown, the bond strength is also significantly lower than that of RelyX Unicem, a self-adhesive luting cement. The bond of Unicem to Cerasmart is also more predictive. The quality of the bond advocated with the use of Ceramir on metal alloys and ceramics, if a true bond exists, appears to apply to a lesser degree to high-density composite materials. Hence, the use of adhesive resin luting cement therefore appears more indicated for luting this new generation of composite materials, in order to take advantage of the adhesive bond rather than a strict mechanical retention. Indeed, a bond can be obtained through the co-polymerization of the add-on composite and the non-converted double bonds of a pre-cured composite resin. Still, due to higher degree of conversion of the high-density CAD/CAM composite blocks such as Cerasmart (Mainjot, 2016), less reactive sites become available as more double bonds are converted, and thus impairs subsequent bonding attempts or repairs (Ruyter 1978, Vankerckhoven 1982). This could potentially lead to lower bonding strength as compared to bonding to direct composite restorations, which have more reactive sites. To this date, limited data is available on the bonding performance of adhesive resin cement to high-density composite blocks, even more so in comparison to bonding performance of the same adhesive resin cement to conventional, direct composite restorations.

The sole use of hydrofluoric acid (HF) as surface treatment has demonstrated to decrease bond strength (Swift 1992, Tate 1993), as opposed to air abrasion with aluminum oxide, which enhanced that bond (Latta 1994). It has been advocated to air abrade and silanate the high-density composite blocks prior to bonding to resin material (Wiegand 2015, Peumans 2016, Fawzy 2008, Nilsson 2000). The use of an intermediate adhesive has been shown to improve bonding performance (Turner 1993). It has been suggested the use of HF, a silane coupling agent and an adhesive treatment of high-density composite materials offers a more predictable outcome than for ceramic materials (El Zohairy 2003). Since glass-ionomer/calcium-aluminate luting cement exhibits a ceramic structure, the use of a silane as coupling agent could prove to enhance the bond between the composite – an organic polymer – and the inorganic glass-ionomer/calcium aluminate cement.

The findings of this study do not rule out the use of a calcium-aluminate/glass ionomer (CA/GI) hybrid cement with high-density composite indirect restorations. As this present study focused on bonding strength, CA/GI cement may not be the luting agent of choice if extra retention is needed due to lack of retentive walls, as the self-adhesive resin cement performed better in that regard. Of all luting cements, adhesive resin has shown to provide the most retention (Zidan 2003). The CA/GI cement did not integrate to the high-density composite hybrid resin, as shown by the 100% adhesive and mixed failure rate. This translates in no added benefit, at least on the interface cement-restorative material. A short-term prospective clinical study has been conducted on cast prosthesis with promising results (Jefferies 2013). However, longer follow-up is needed in order to rightfully assess the performance of this luting cement.

On the other hand, CA/GI luting cement may prove to be of significant benefit in patient at high-risk of caries. If caries prevention is proven to be true, aging patients whose hygiene may be deficient either due to alteration in salivary flow or lack of motor skills may benefit from the increased resistance to acidity. The basic pH helps prevent demineralization from acidity coming from the bio-film and a cariogenic diet. Patients whose prosthesis margins sit on dentin rather than enamel can also benefit from CA/GI cementation: higher pH and phosphate uptake from oral fluids promote the formation of an apatite seal, hence preventing microleakage. Microleakage is a well-documented complication of adhesive bonding agents as the bond strength is compromised by water sorption, which affects physical properties of the adhesive.

The vast majority of Ceramir samples failed at the interface between the substrate and the cement. This adhesive failure is in line with what has been previously observed with luting agents on polymeric CAD/CAM materials (Basler 2011).

Limitations

Numerous samples within the Ceramir groups debonded before testing. Some of the cement buttons mounted, as many as eight, debonded before or during the incubation period. When the separation occurred prior, the buttons did not resist the withdrawal of the mold after the setting time. When it occurred during the incubation, the buttons were seen floating on the surface of the PBS solution, most likely due to lack of adherence of the buttons to the bonding surface. When the debonding was observed prior to the incubation period, failed samples were reinserted in the bonding clamp and cement buttons remounted. Otherwise, mean values of bond strength obtained for the Ceramir groups would have been lower. One can extrapolate that such a large proportion of failure within one group shows a lack of adherence to the substrate, therefore not suitable as a bonding agent. However, there is no way to determine how significant this would have been as these failed specimens were not recorded as “zero” in terms of bond strength.

This present study has many limitations. Voids were observed within the luting cement, which, when located at the interface between the cement and the substrate, could have introduced variability in the contact surface area and negatively affected the bond strength. These were observed more frequently among Ceramir samples than Unicem ones. In some cases, the cement extruded outside the mould at the interface, which could have positively affected the bond strength by increasing the contact surface area.

In this study, phosphate-buffered saline (PBS) solution was used as the preferred environment to store the specimens as it comes closest to the oral environment. It is claimed by the manufacturer that the calcium-aluminate in Ceramir takes up the phosphate from the oral fluids to form apatite crystals, hence promoting integration to the tooth structure. However, exposure of the glass ionomer component to excess amount of water is known to negatively affect the physical properties and integrity of the glass ionomer component, especially in the early phase as it is highly susceptible to dissolution, as discussed earlier. Furthermore, it is well known that water storage influences negatively the physical properties of resin blocks (Ruse et Sadoun, 2014) and the bond strength between resin blocks and resin cement (Higashi 2016). However, it is not known whether PBS affects one way or the other the interface Ceramir/composite block, or if it affects it at all.

Cerasmart blocks were not surface-treated before Ceramir was applied to them. Preconditioning of the Cerasmart blocks with 50-micron aluminum oxide sand-blasting - as the protocol for luting with resin cement warrants - could have increased the substrate's wettability and surface area, thus increased mechanical retention. This is believed to have a positive effect on bond strength (Higashi 2016).

The bonding strength obtained in this study for the RelyX Unicem is relatively low compared to ideal values advocated for composite resin bonding (15 to 25 MPa). This could be explained by the absence in the protocol of the use of a silane coupling agent on the substrate's surface after sandblasting with 50-micron aluminum oxide particles, as recommended by the manufacturer. Studies have reported the high degree of conversion achieved with HT/HP polymerization prevents effective bonding without prior conditioning (Bähr et al 2013). On the other hand, sandblasting did improve the bonding (Basler 2011). Sandblasting combined with silanization produced higher bond strength than sandblasting or salinization alone (Higashi 2016).

CONCLUSION

Based on the result of this study, it appears to the author that a hybrid of calcium-aluminate/glass ionomer may not be the cement of choice for luting high-density composite restorations when preparation design cannot provide predictable retention, and adhesive resin luting cement is readily available. This however does not preclude its use. Based on existing literature, there may be some other benefits using CA/GI cement, such as prevention of microleakage with the formation of apatite, and caries prevention, due to high pH values, among others. In other words, there is no universal cement that is indicated for all clinical situations: the provider still has to use his clinical judgment to determine which cement is best suited for each clinical situation.

In a situation where CA/GI is indicated as a luting agent for the aforementioned reasons, it may be relevant to conduct further studies on which surface treatment would provide the highest mechanical retention. The most commonly encountered surface treatments in the literature –

regardless of the material used - are 37%-concentration of phosphoric acid etching, 5 to 10%-concentration hydrofluoric acid etching, diamond-bur roughening, aluminum-oxide particle air abrasion, use of a silane coupling agent, tribochemical treatment, or a combination of. Testing different surface treatment could prove valuable to downplay the lack of bonding offered by CA/GI luting cement to high-density composite materials.

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APPENDIX A

ANOVA Analysis

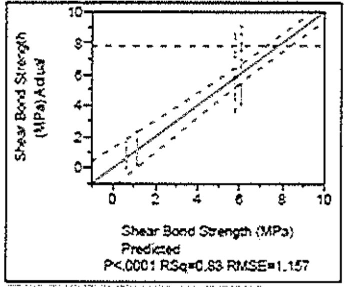
JMP_AddIn - Fit Least Squares

Page 1 of 2

Response Shear Bond Strength (MPa)

Whole Model

Actual by Predicted Plot



Summary of Fit

RSquare	0.832513
RSquare Adj	0.825813
Root Mean Square Error	1.157012
Mean of Response	3.426582
Observations (or Sum Wgts)	79

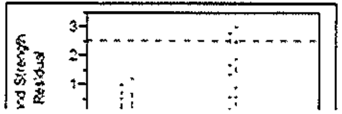
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	499.05334	166.351	124.2652
Error	75	100.40084	1.339	Prob > F
C. Total	78	599.45418		<.0001*

Parameter Estimates

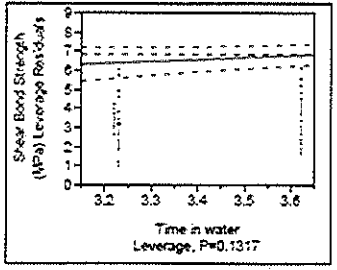
Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	3.3915789	0.130206	26.05	<.0001*
Time in water[1-Day]	-0.198421	0.130206	-1.52	0.1317
Cement[Unicem]	2.5084211	0.130206	19.26	<.0001*
Time in water[1-Day]*Cement[Unicem]	0.0584211	0.130206	0.45	0.6550

Residual by Predicted Plot



Time in water

Leverage Plot



Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
1-Day	3.1931579	0.18533121	3.25897
7-Day	3.5900000	0.18293974	3.59000

LSMeans Differences Student's t

α= 0.050 t= 1.9921

LSMean[i]	LSMean[j] Mean[i]-Mean[j] Std Err Dif Lower CL Dif Upper CL Dif	1-Day	7-Day
	1-Day	0 0 0 0	-0.3968 0.26041 -0.9156 0.12193
	7-Day	0.39684 0.26041 -0.1219 0.91561	0 0 0 0

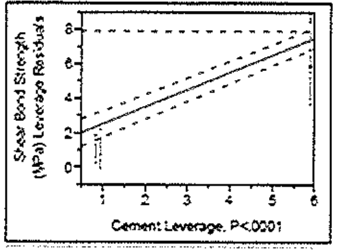
Level		Least Sq Mean
7-Day	A	3.5900000
1-Day	A	3.1931579

Levels not connected by same letter are significantly different.

Time in water*Cement

Cement

Leverage Plot



Least Squares Means Table

Level	Least Sq Mean	Std Error	Mean
Unicem	5.9000000	0.18293974	5.90000
Ceramir	0.8831579	0.18533121	0.85974

LSMeans Differences Student's t

α= 0.050 t= 1.9921

LSMean[i]	LSMean[j] Mean[i]-Mean[j] Std Err Dif Lower CL Dif Upper CL Dif	Unicem	Ceramir
	Unicem	0 0 0 0	5.01684 0.26041 4.49807 5.53561
	Ceramir	-5.0168 0.26041 -5.5356 -4.4981	0 0 0 0

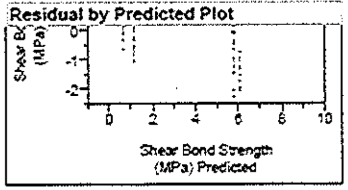
Level		Least Sq Mean
Unicem	A	5.9000000
Ceramir	B	0.8831579

Levels not connected by same letter are significantly different.

Response Shear Bond Strength (MPa)

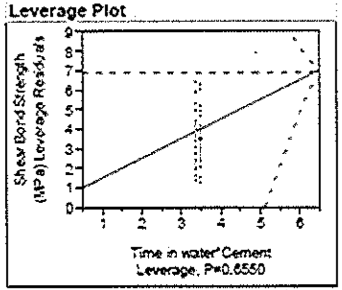
Whole Model

Residual by Predicted Plot



Time in water*Cement

Leverage Plot



Least Squares Means Table

Level	Least Sq Mean	Std Error
1-Day,Unicem	5.7600000	0.25871586
1-Day,Ceramir	0.6263158	0.26543687
7-Day,Unicem	6.0400000	0.25871586
7-Day,Ceramir	1.1400000	0.25871586

LSMeans Differences Tukey HSD

α= 0.050 Q= 2.62758

LSMean[j]

LSMean[i]

Mean[i]-Mean[j] Std Err Dif Lower CL Dif Upper CL Dif	1-Day,Unicem	1-Day,Ceramir	7-Day,Unicem	7-Day,Ceramir
1-Day,Unicem	0 0 0 0	5.13368 0.37066 4.15974 6.10763	-0.28 0.36588 -1.2414 0.68138	4.62 0.36588 3.65862 5.58138
1-Day,Ceramir	-5.1337 0.37066 -6.1076 -4.1597	0 0 0 0	-5.4137 0.37066 -6.3876 -4.4397	-0.5137 0.37066 -1.4876 0.46026
7-Day,Unicem	0.28 0.36588 -0.6814 1.24138	5.41368 0.37066 4.43974 6.38763	0 0 0 0	4.9 0.36588 3.93862 5.86138
7-Day,Ceramir	-4.62 0.36588 -5.5814 -3.6586	0.51368 0.37066 -0.4603 1.48763	-4.9 0.36588 -5.8614 -3.9386	0 0 0 0

Level			Least Sq Mean
7-Day,Unicem	A		6.0400000
1-Day,Unicem	A		5.7600000
7-Day,Ceramir		B	1.1400000
1-Day,Ceramir		B	0.6263158

Levels not connected by same letter are significantly different.

APPENDIX B

Weibull analysis

Pf = 1 - e^[-(σ/σ0)^m]

Characteristic strength = strength occurred at a probability of failure of 63.2%

		Sorted Unicem, 1-Day				Sorted Ceramir, 1-Day			
	i	σ (MPa)	ln (σ)	Pf (CAD-I)	ln{ln[1/(1-Pf)]}	σ (MPa)	ln (σ)	Pf (CAD-I)	ln{ln[1/(1-Pf)]}
	1	3.5	1.253	0.025	-3.676	0.0E+00		0.026	-3.624
	2	3.7	1.308	0.075	-2.552	0.0E+00		0.079	-2.498
	3	4.3	1.459	0.125	-2.013	0.0E+00		0.132	-1.958
	4	4.5	1.504	0.175	-1.648	0.0E+00		0.184	-1.592
	5	4.6	1.526	0.225	-1.367	0.0E+00		0.237	-1.308
	6	4.7	1.548	0.275	-1.134	0.3	-1.204	0.289	-1.074
	7	4.8	1.569	0.325	-0.934	0.5	-0.693	0.342	-0.871
	8	5.1	1.629	0.375	-0.755	0.6	-0.511	0.395	-0.689
	9	5.3	1.668	0.425	-0.592	0.7	-0.357	0.447	-0.522
	10	5.4	1.686	0.475	-0.440	0.7	-0.357	0.500	-0.367
	11	5.6	1.723	0.525	-0.295	0.7	-0.357	0.553	-0.218
	12	5.7	1.740	0.575	-0.156	0.8	-0.223	0.605	-0.073
	13	5.9	1.775	0.625	-0.019	0.8	-0.223	0.658	0.070
	14	6.3	1.841	0.675	0.117	0.8	-0.223	0.711	0.215
	15	6.3	1.841	0.725	0.255	0.9	-0.105	0.763	0.365
	16	7.1	1.960	0.775	0.400	1.1	0.095	0.816	0.526
	17	7.4	2.001	0.825	0.556	1.1	0.095	0.868	0.707
	18	8.2	2.104	0.875	0.732	1.3	0.262	0.921	0.932
	19	8.3	2.116	0.925	0.952	1.6	0.470	0.974	1.291
	20	8.5	2.140	0.975	1.305				
	MEAN	5.760							
	SD	1.492							
Weibull Modulus	m	4.6676				m	1.5987		
	b	-8.5893				b	0.4012		
Characteristic Strength	e^b/m	6.2978				e^b/m	0.7781		

Sorted Unicem, 7-Day σ (MPa)	ln (σ)	Pf (CAD-I)	ln{ln[1/(1-Pf)]}	Sorted Ceramir, 7-Day σ (MPa)	ln (σ)	Pf (CAD-I)	ln{ln[1/(1-Pf)]}
4.0	1.386	0.025	-3.676	0.1	-2.303	0.025	-3.676
4.1	1.411	0.075	-2.552	0.3	-1.204	0.075	-2.552
4.3	1.459	0.125	-2.013	0.4	-0.916	0.125	-2.013
4.5	1.504	0.175	-1.648	0.5	-0.693	0.175	-1.648
4.7	1.548	0.225	-1.367	0.6	-0.511	0.225	-1.367
4.7	1.548	0.275	-1.134	0.8	-0.223	0.275	-1.134
4.8	1.569	0.325	-0.934	0.8	-0.223	0.325	-0.934
5.0	1.609	0.375	-0.755	0.9	-0.105	0.375	-0.755
5.1	1.629	0.425	-0.592	1.1	0.095	0.425	-0.592
5.3	1.668	0.475	-0.440	1.1	0.095	0.475	-0.440
6.3	1.841	0.525	-0.295	1.2	0.182	0.525	-0.295
6.4	1.856	0.575	-0.156	1.3	0.262	0.575	-0.156
6.6	1.887	0.625	-0.019	1.4	0.336	0.625	-0.019
6.9	1.932	0.675	0.117	1.5	0.405	0.675	0.117
7.5	2.015	0.725	0.255	1.5	0.405	0.725	0.255
7.6	2.028	0.775	0.400	1.7	0.531	0.775	0.400
7.6	2.028	0.825	0.556	1.7	0.531	0.825	0.556
7.9	2.067	0.875	0.732	1.8	0.588	0.875	0.732
8.5	2.140	0.925	0.952	1.8	0.588	0.925	0.952
9.0	2.197	0.975	1.305	2.3	0.833	0.975	1.305

m	1.6154	m	4.4578
b	-0.4561	b	-8.4359
e^b/m	1.3262	e^b/m	6.6352

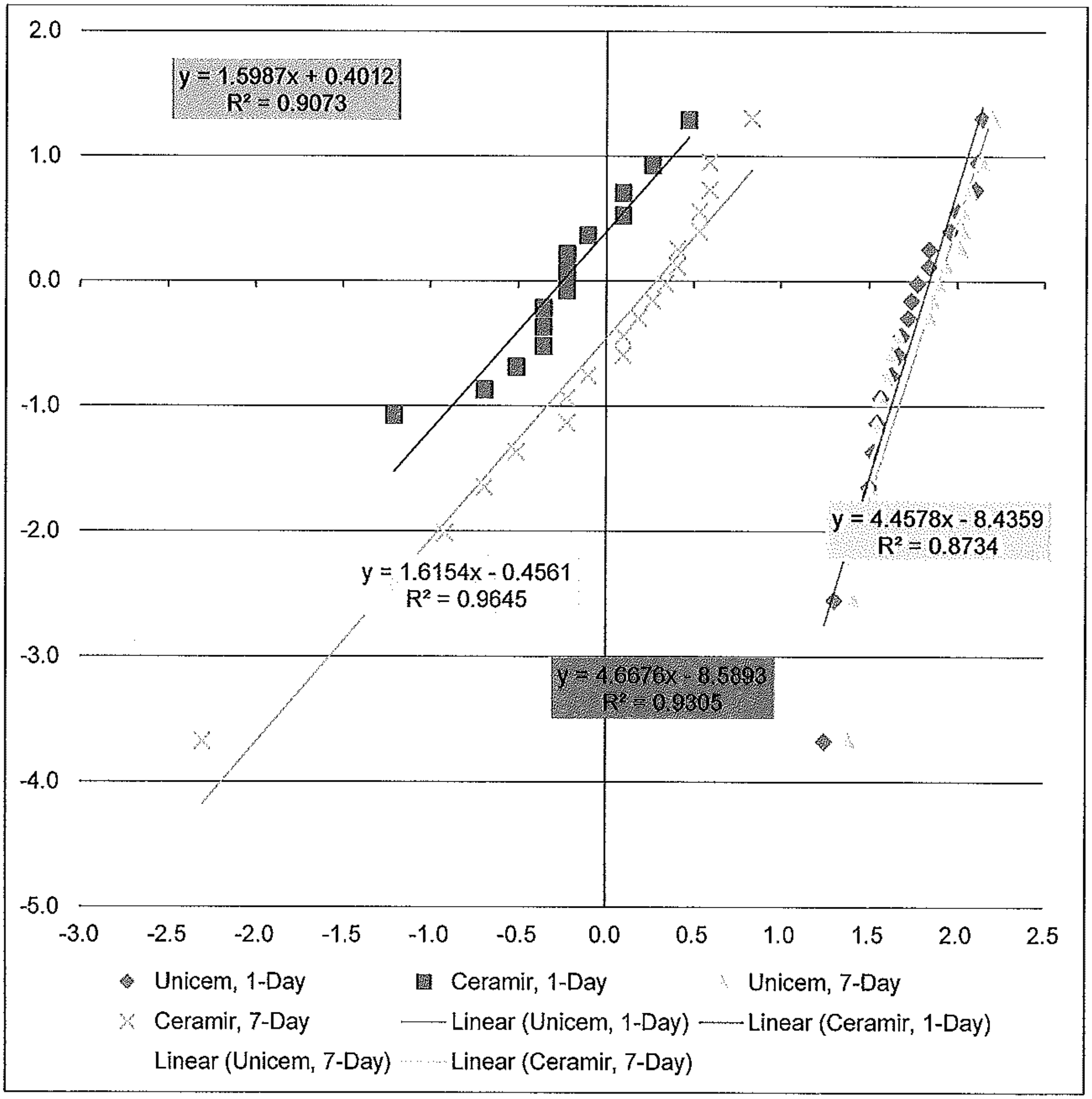
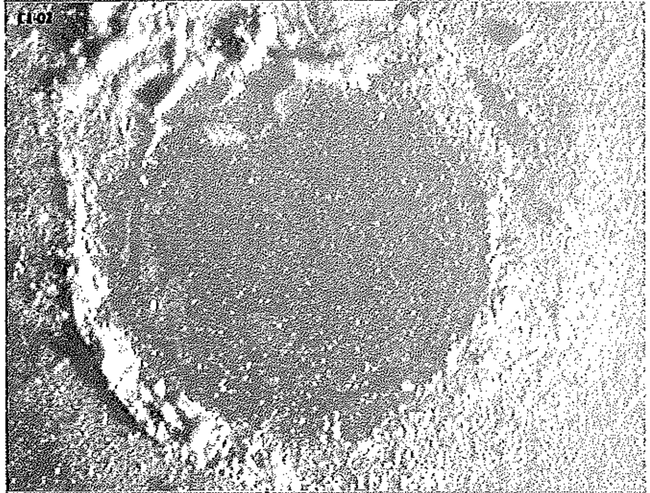
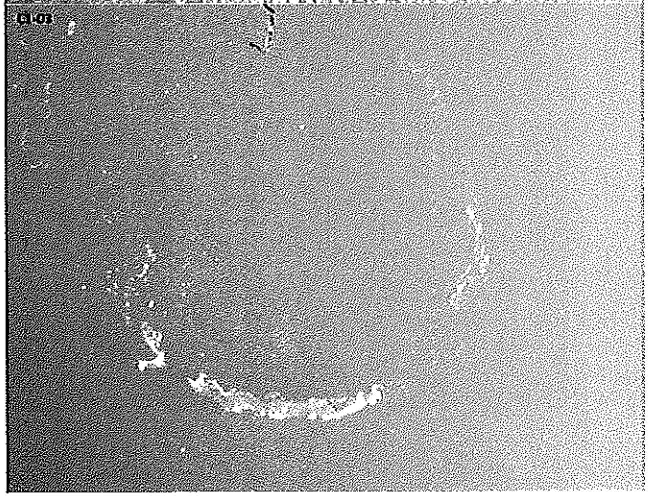
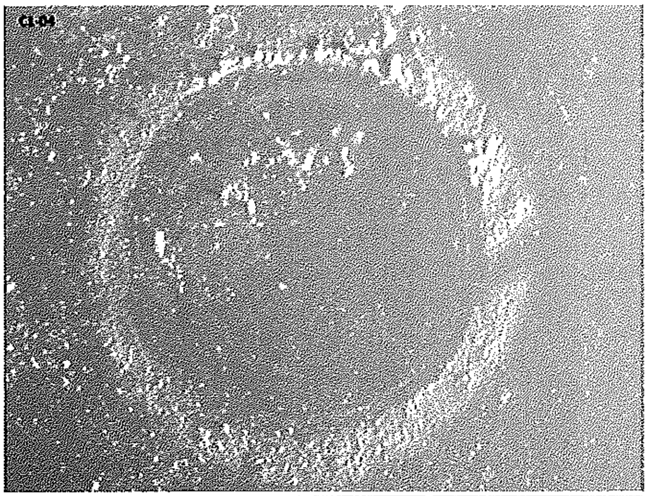


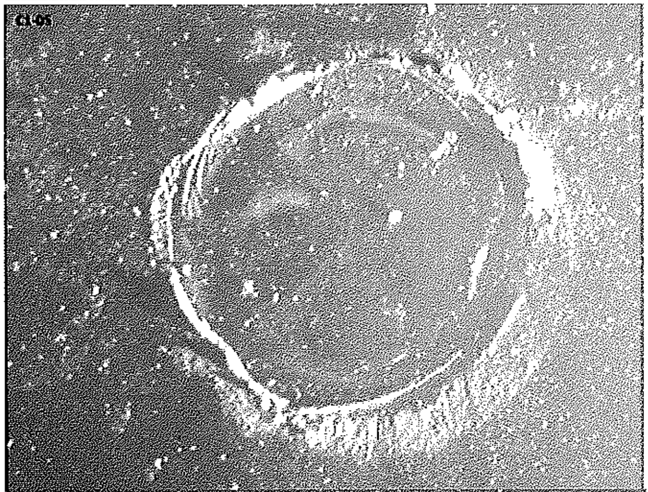
Figure 2: Weibull modulus (slope) for RelyX Unicem and Ceramir C&B at 1- and 7-day periods. Unicem groups displays higher Weibull moduli than Ceramir groups. Hence, the strength of Unicem is more predictable than Ceramir.

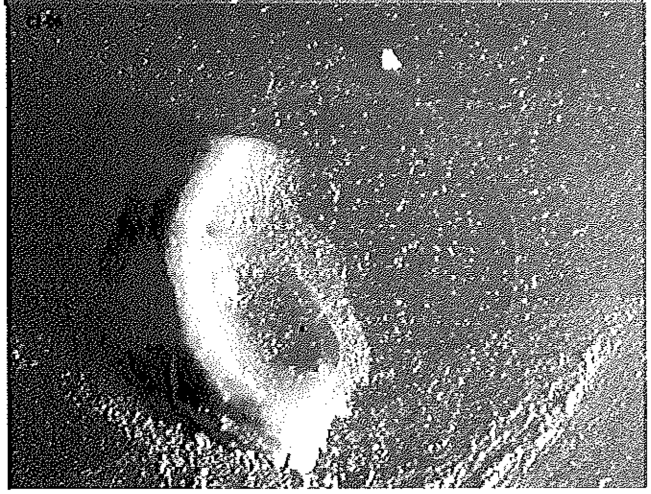
APPENDIX C

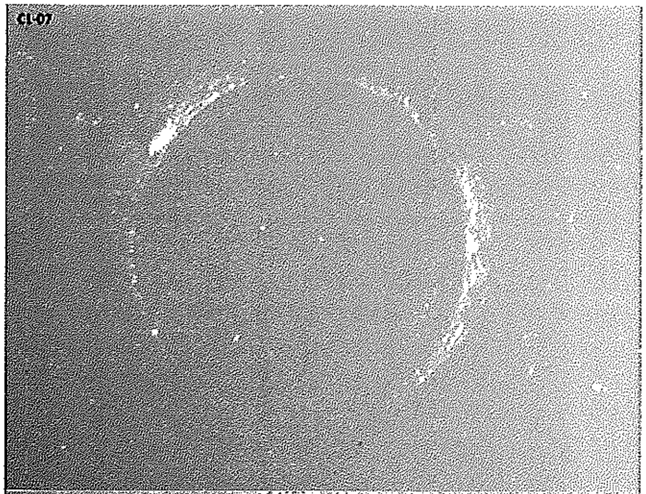








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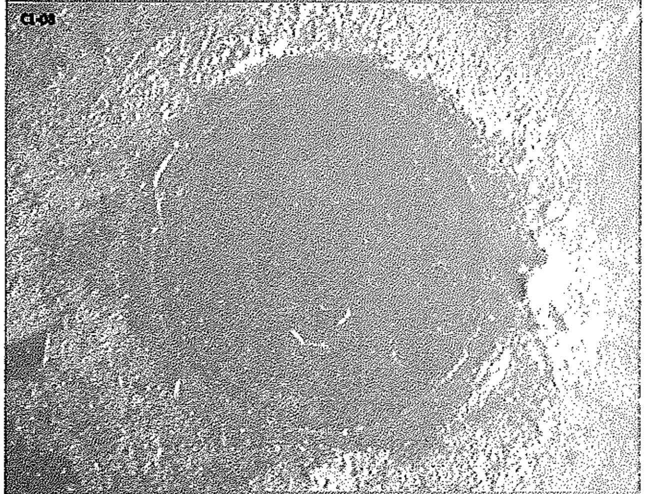
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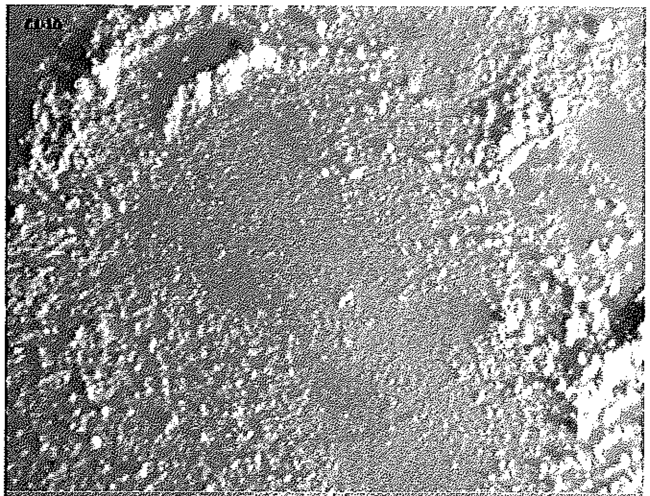
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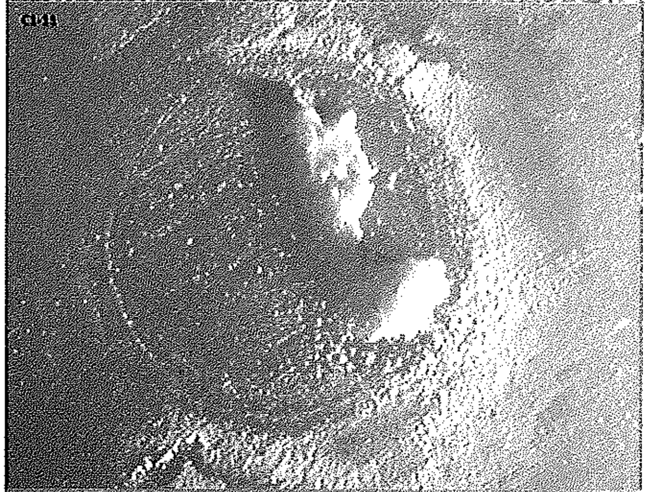
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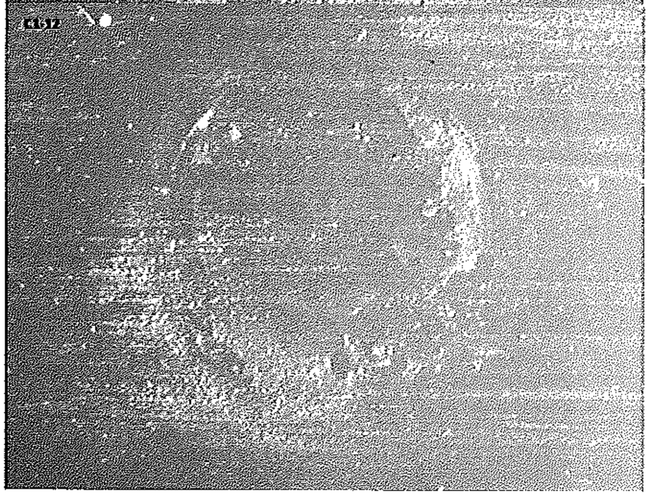
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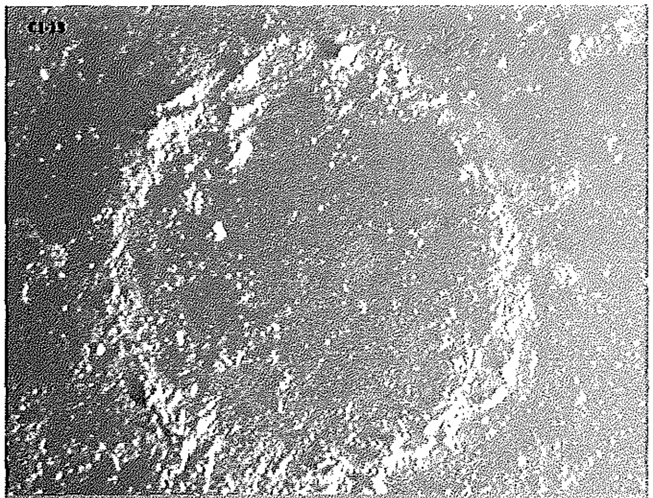
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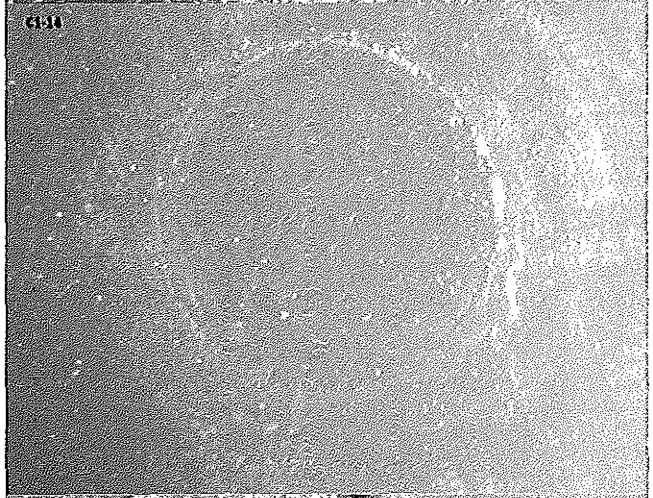
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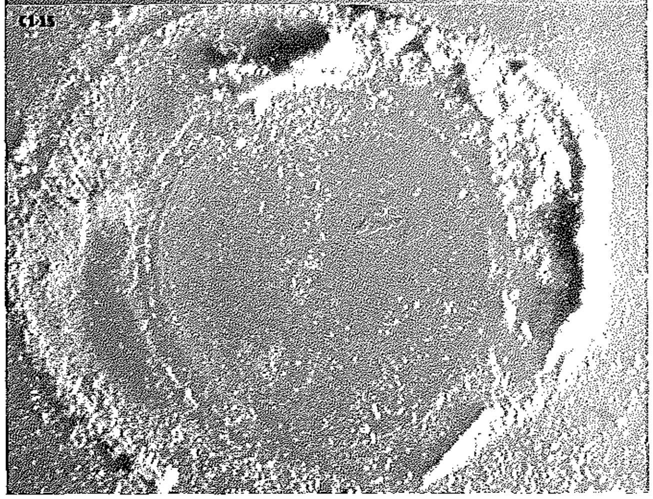
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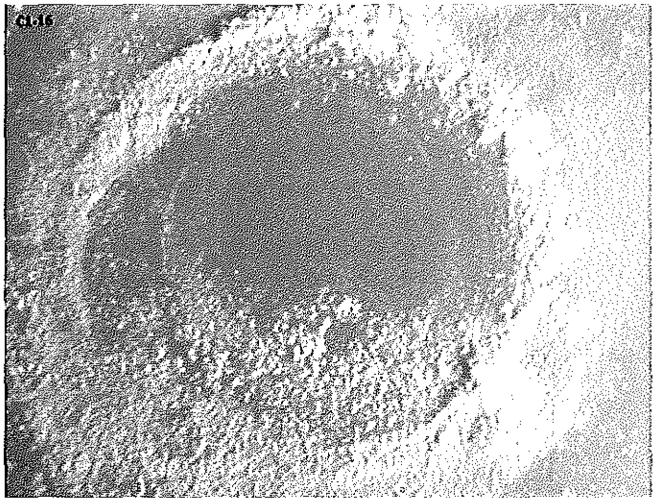
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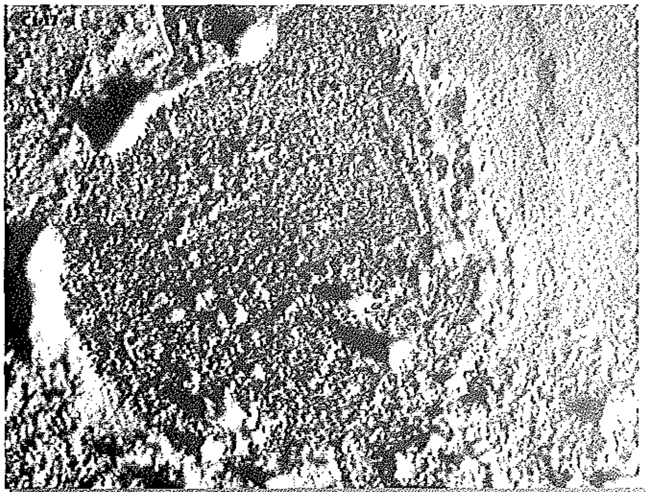
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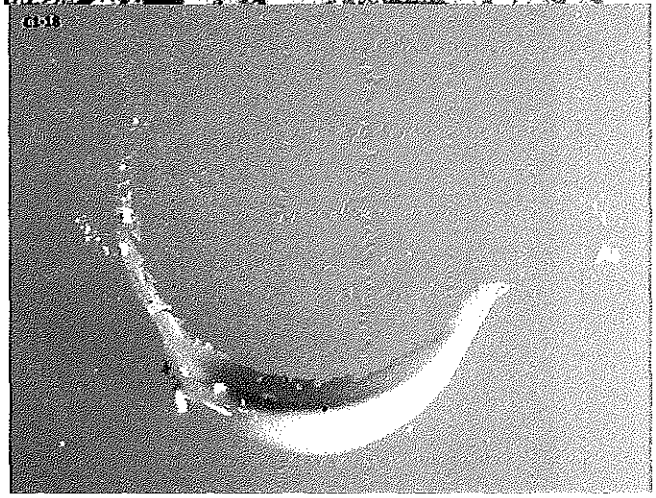
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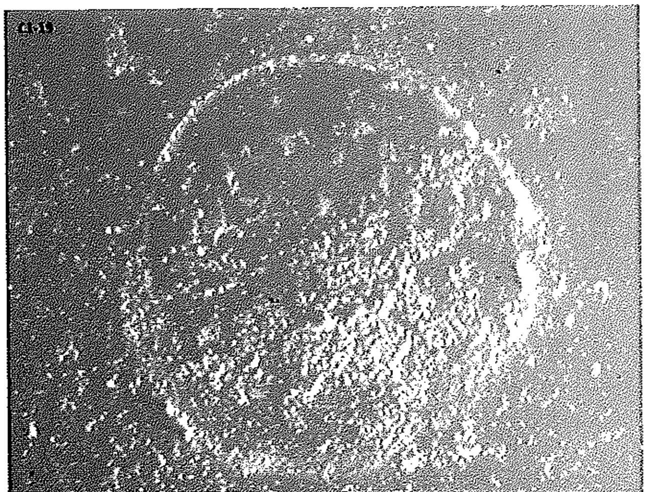
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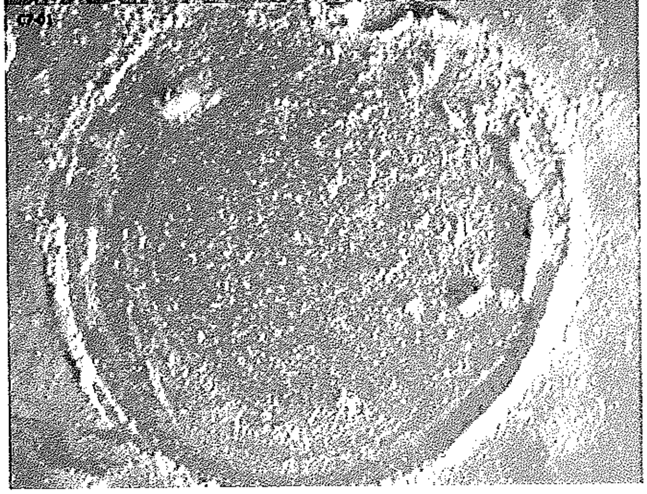
C1-20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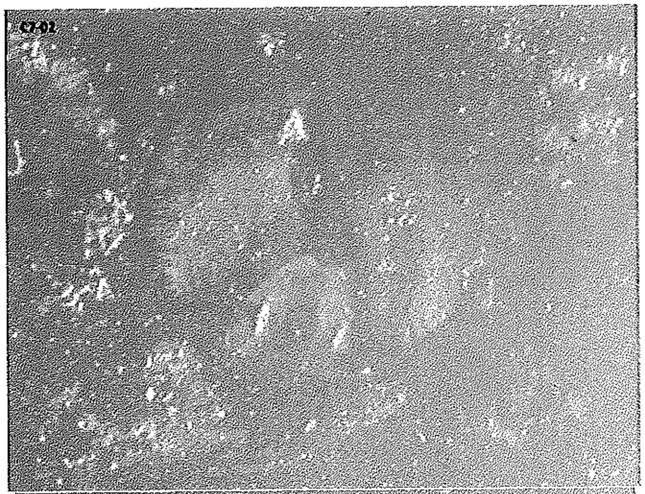
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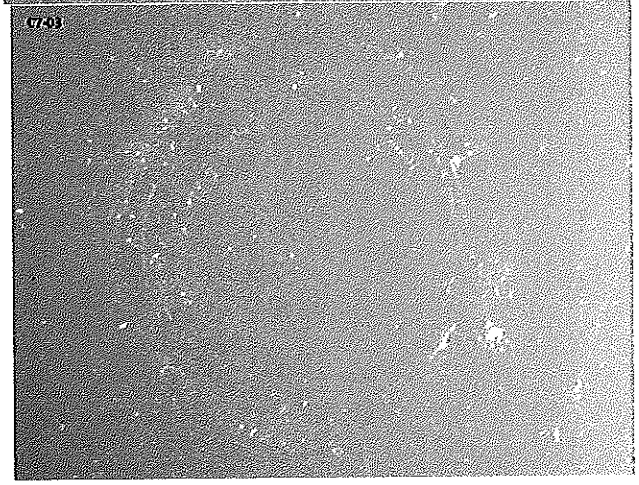
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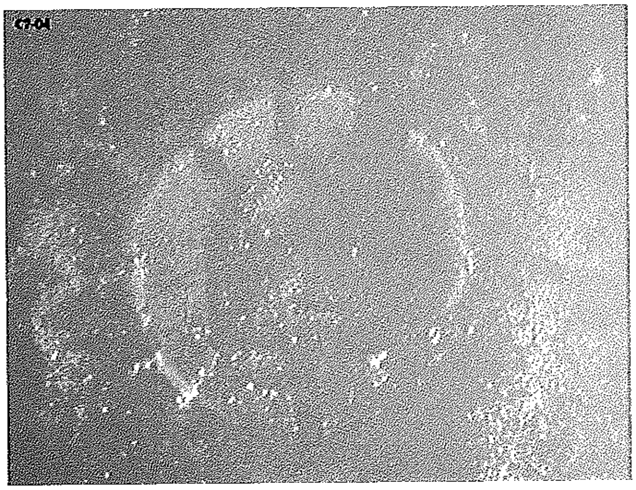
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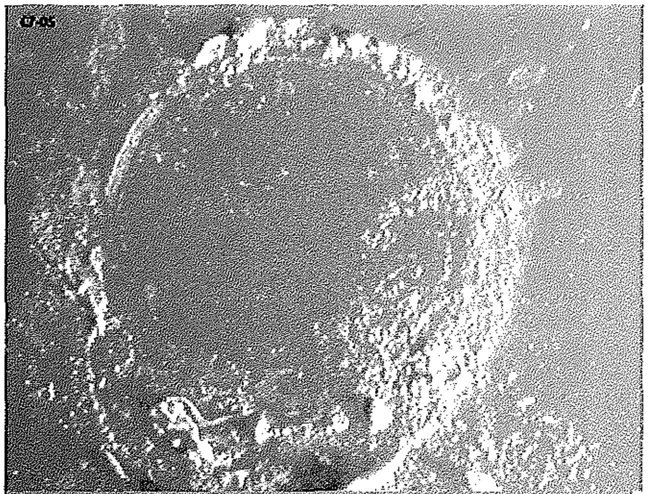
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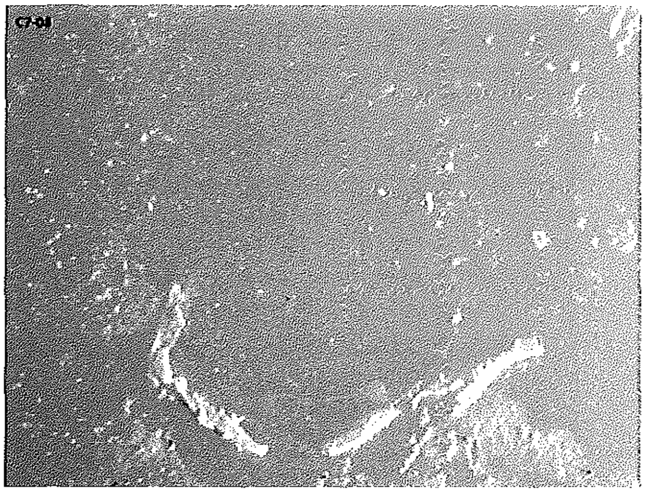
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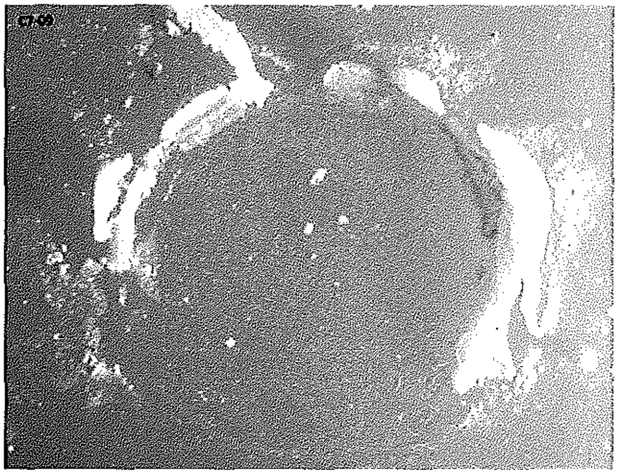
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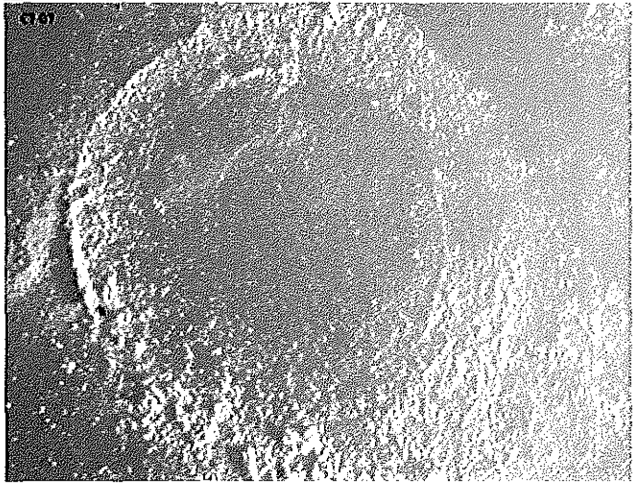
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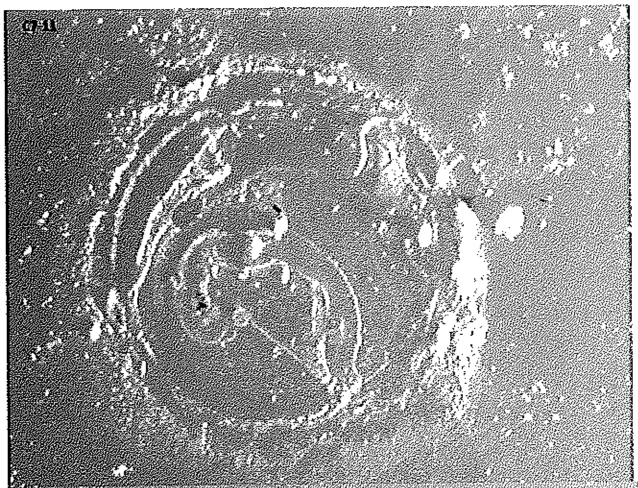
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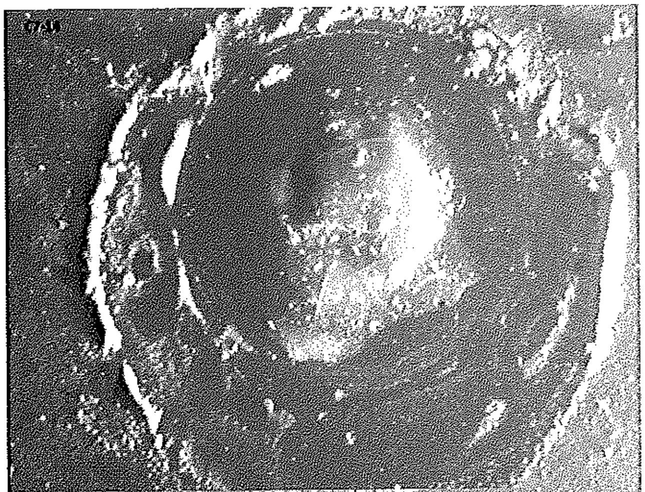
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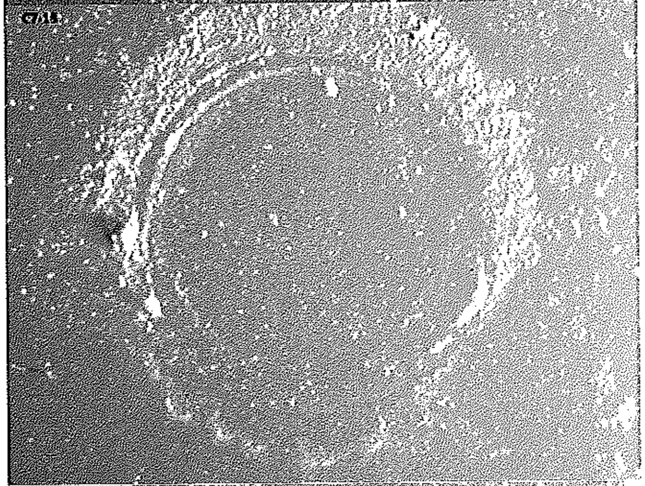
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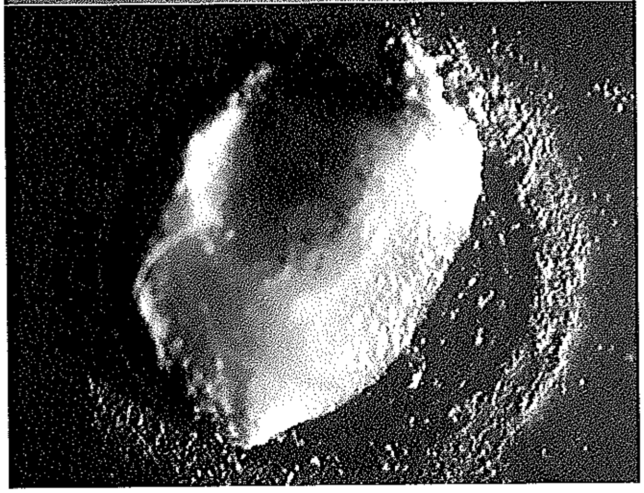

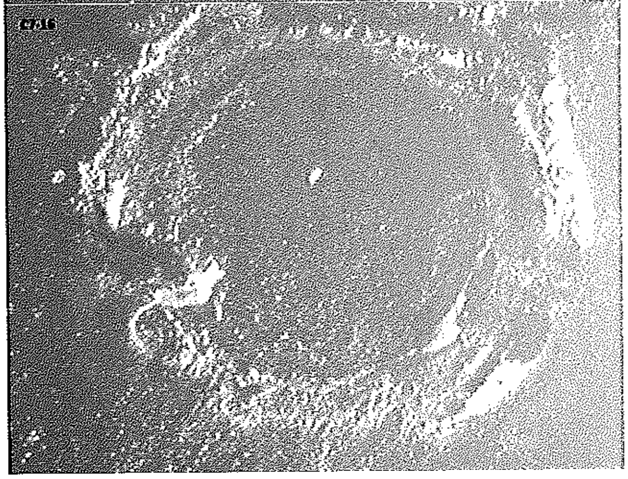
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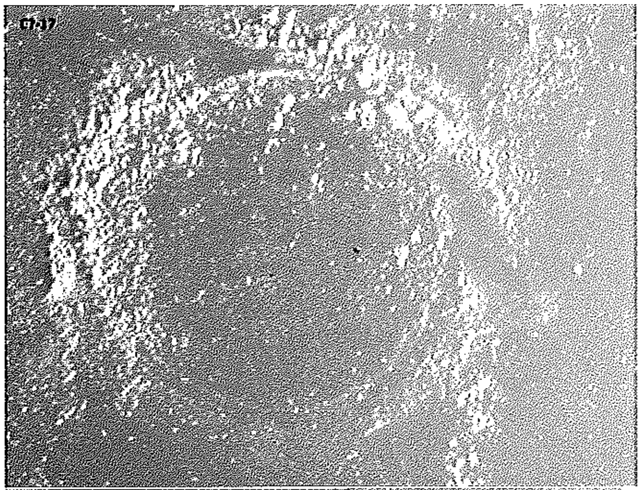
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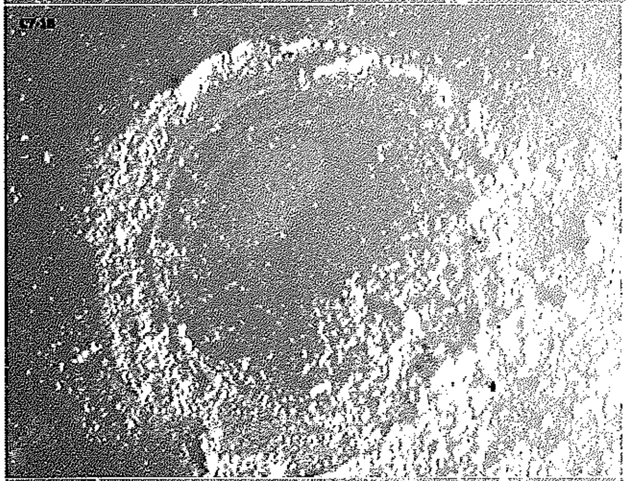
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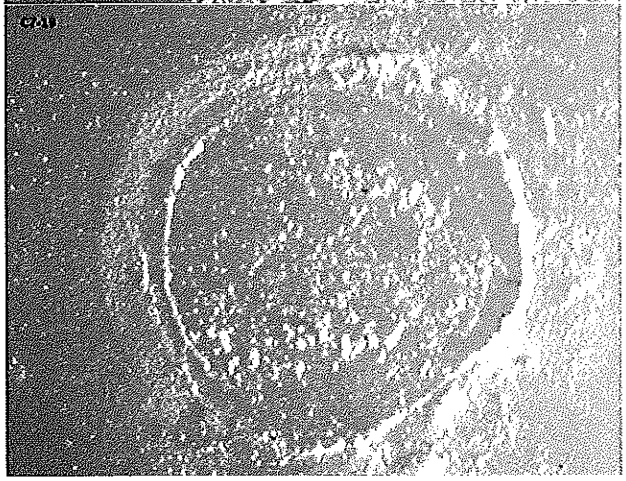
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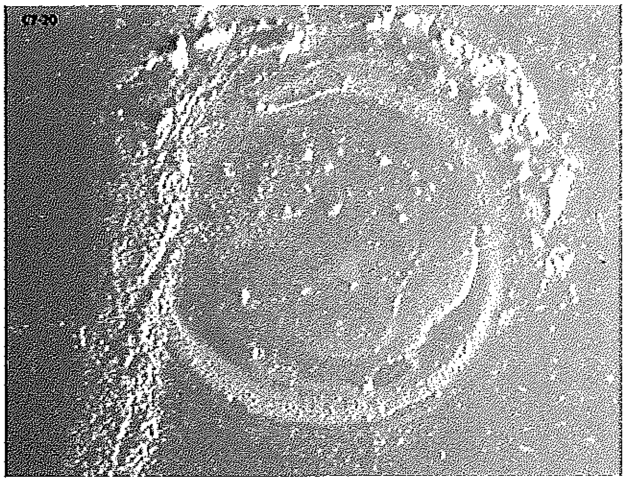
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