# Laboratory Testing Protocol for Evaluation of Restoration Polishing Systems

## Scope

This protocol for the laboratory testing of restoration polishing systems was developed to provide clinically relevant laboratory information that will be used as part of a report on these products in a future issue of the *ADA Professional Product Review*. The tests described in this protocol should provide the dental practitioner with appropriate comparative scientific information to assist in the purchase of restoration polishing systems.

## Reference Documents:

1. ANSI/ADA Specification 27, "Resin-Based Filling Materials," July 2005.
2. ISO/DIS 4049 "Dentistry – Polymer-based filling, restorative and luting materials," 2008-02-28.
3. ISO 10650 "Dentistry – Powered Polymerization Activators".
4. C. S. Jones, R. W. Billington, and G. J. Pearson, "Laboratory study of the loads, speeds and times to finish and polish direct restorative materials," *Journal of Oral Rehabilitation*, **2005** 32: 686-692.
5. C. S. Jones, R. W. Billin gton, and G. J. Pearson, "The in vivo perception of roughness of restorations," *British Dental Journal*, **2004** 196: 42-45.
6. Juliana Da Costa, Jack Ferracane, Rade Paravina, Rui Fernando Mazur, and Leslie Roeder, "The Effect of Different Polishing Systems on Surface Roughness and Gloss of Various Resin Composites," *Journal of Esthetic Restorative Dentistry*, **2007** 19: 214-226.
7. Tamayo Watanabe, Masashi Miyazaki, and B. Keith Moore, "Influence of polishing instruments on the surface texture of resin composites," *Quintessence International*, January **2006** 37 Number 1: 61-67.
8. ASTM D 523 – 89, "Standard Test Method for Specular Gloss." Annual *Book of ASTM Standards*, **Volume 6.01**, "Paint - Tests for Chemical, Physical, and Optical Properties; Appearance".
9. ASTM D 3980, "Practice for Interlaboratory Testing of Paint and Related Materials." Annual *Book of ASTM Standards*, **Volume 6.01**, "Paint - Tests for Chemical, Physical, and Optical Properties; Appearance".
10. Quirynen M., Bollen C.M., Papaioannou W., et al., "The influence of titanium abutment surface roughness on plaque accumulation and gingivitis: short-term observations," Int. J. Oral Maxillofac Implants 1996; 11:169-78.

## General Requirements:

1. Two different types of composite materials shall be used with the polishing systems in this study: Heliomolar (microfill composite) and Esthet-X HD (hybrid, mini-fill).
2. The A2 Shade shall be used for all of the composite materials in this protocol.
3. For the tests in this protocol where water is referenced, deionized water shall be used in all instances.

## Materials:

**Teflon molds**, for the preparation of cylindrical specimens, 4 mm long x 15 mm in diameter. Note that the molds shall be vented such that when pressure is applied to the molds excess material can flow out the vents.

**Two glass plates**, of sufficient area to cover one side of the mold.
**White filter paper**.
**Film**, transparent to the activating radiation, e.g., polyester, 50 ± 30 μm thick.
**External Energy Source**. In these tests, the Optilux 501 (Kerr Corporation), with a 13 mm tip, shall be used as the external energy source. The light intensity of the external energy source shall be measured, according to ISO 10650 "Dentistry–Powered Polymerization Activators," and recorded. If any of the intensity readings vary significantly from previous readings (e.g., ±50 mW/cm$^2$), the external energy source shall be investigated and repaired or replaced before further testing is performed. Also, the intensity shall be above 300 mW/cm$^2$.
**Dial indicator,** accurate to 0.001 mm.
**20 lbs press**3in diameter contact area
**Garolite, grade G-10**, 15 mm in diameter.
**Cyanoacrylate glue** (e.g., Loctite 414, Loctite, Corp.)
**Surface Profilometer** (e.g., Surtronic 3+, Taylor Hobson, Inc.).
**Diamond finishing burs** (e.g., Midwest Diamonds, fine grit, ISO 806 314 158 514 014).
**Custom-built polishing apparatus**. The apparatus shall allow a polishing disk to be applied to the prepared sample. Aspects of the application that shall be controlled are, contact angle, polishing system rpm, contact time, linear traversing speed, application pressure (normal to the surface of the prepared sample).
**Custom-built initial sample roughness preparation apparatus.** The apparatus shall allow an initial roughness to be prepared on the surface of a composite sample that has been prepared in accordance with this protocol. The apparatus shall have a flat turntable capable of spinning at a rate of 200 RPM. The specimen holder shall have the capability of rotating at a rate of <insert rate here> as well as the ability to lower the sample into the turntable at a user defined distance and speed. The apparatus shall include a water spray and shall be capable of producing an average Ra value on a composite sample of 0.9 (+/- 0.1) um, measured in accordance with this protocol.
**Glossmeter** (e.g., Novo-Curve Glossmeter, Rhopoint Instrumentation LTD, East Sussex, UK).

## Procedure:

1. **Preparation of test specimens**.
    a. Test samples shall be prepared at laboratory temperature and humidity (approx. 23°C and 50% humidity).
    b. Cover one of the glass plates with the polyester film, and position the mold upon it.
    c. Prepare the material in accordance with the manufacturer's instructions and fill the mold to a slight excess with the material (place the material as evenly as possible taking care to exclude air bubbles or voids).
    d. Place a second piece of polyester film onto the material in the mold and cover this with the second glass plate. Apply 20lbs of pressure using a hydraulic press for a minimum of 30 seconds to displace any excess material (note that the molds shall be vented such that when pressure is applied to the molds excess material can flow out the vents).
    e. Remove the glass plate (keep the polyester film in place) and place the exit window of the external energy source against the glass slide and irradiate that section of the specimen for the recommended exposure time.

f. Move the exit window of the external energy source to the section next to the previous one, overlapping the previous section by half the diameter of the exit window, and irradiate for the appropriate time. Continue this procedure until the entire specimen has been irradiated for the recommended exposure time.
g. Replace the glass plate, turn the mold over, and remove the bottom glass plate (keep the polyester film in place) and place the exit window of the external energy source against the glass slide and repeat the irradiation procedure on the bottom side of the specimen.
h. After the irradiation procedure is complete, remove the specimen from the mold and place a drop of cyanoacrylate glue on the side of the specimen which was in contact with the 20lbs load to attach the specimen to a 15 mm diameter Garolite rod, making sure that the specimen and the rod are concentric. Note that the face of the Garolite rod shall be machined flat such that the deviation across the face is less than ±0.01 mm. Furthermore, the face of the Garolite rod shall be sprayed with primer before the cyanoacrylate glue is dropped on the surface. Also, one region of the rod shall be ground flat lengthwise to provide a reference point for mounting of the specimen in the polishing, roughness, and height measuring devices.
i. For each polishing system manufacturer, prepare five specimens in this manner for each type of composite material. Therefore, for each polishing systems, since two types of composite materials shall be evaluated, a total of ten specimens shall be prepared for evaluation.

2. **Preparation of initial surface roughness**. After the specimen has been appropriately mounted to the Garolite rod place the specimen in the initial roughness preparation apparatus. Once the specimen is mounted properly in the apparatus the specimen shall be lowered such that it comes in contact with the 120 grit silicon carbide paper mounted to the turntable of the apparatus. This shall be the intital position. The turntable shall then be rotated at 200 rpm and the specimen shall be rotated, counter to the rotation of the turntable, at a speed of <insert speed here>. The sample shall then be lowered into the paper at an acceleration of 0.5mm/s/s with a trapezoidal speed curve in 0.01mm increments up to 0.10mm (Helimolar) or 0.15mm (Esthet-X) total distance from the initial position holding at each position for 7 seconds. Then the specimen will be immediately raised clear of the turntable and the rotation of the specimen will be reversed such that the turntable and the specimen are rotating in the same direction. The specimen will then be lowered to 0.05mm from the final position of the previous lowering sequence at an acceleration of 0.5mm/s/s with a trapezoidal speed curve. The specimen shall be held at that position for 7 seconds. The specimen will then immediately be raised such that it no longer contacts the carbide paper and the apparatus shall be turned off. The specimen shall be removed and dried with tissue paper and the average Ra value on the surface of the specimen shal be verified to 0.9 (+/- 0.1) um, measured in accordance with this protocol. If the initial average roughness in not within this window the specimen shall be remounted in the initial roughness apparatus and the preparation of initial surface roughness will again be performed.
3. **Measurement of initial height of specimen**. With the flat region of the Garolite rod as the reference point (0 degree position), zero the dial indicator at the 0 degree reference point and measure the deviation in height at 60 degree

increments along the circumference of the specimen (1 mm in from the outer diameter) **NOTE: After the finishing step and the f**inal polishing step, the specimen shall be mounted back on the mechanical breadboard and the same points shall be checked and the change in heights recorded.

**Gloss Measurements**. After the initial height of the specimen is recorded, gloss measurements shall be taken using a glossmeter with a 60° geometry. The glossmeter shall be calibrated according to ASTM D523. The specimen shall be mounted such that the designated control region is centered over the aperture opening of the glossmeter. Three readings shall be taken on the surface of the polished specimen and the mean shall be reported. If the range of the three gloss readings is greater than 2 gloss units, then additional readings shall be taken and the mean shall be calculated after discarding divergent results in accordance with the section on outliers in ASTM D3980. The specimen shall then be positioned such that the test (polish) region is centered directly over the aperture of the glossmeter and three measurements shall be taken in the same fashion as stated previously.

4. **Measurement of surface roughness**.
    a. The Surtronic 3+ shall be used to measure the surface roughness, with the following settings: 2.5 mm traverse length, cut-off value of 0.25 mm, and traverse speed of 1 mm/s. A filter parameter of 0.25 shall be used.
    b. The specimen shall be mounted such that the flat region of the Garolite rod is perpendicular to the path of the stylus. This position shall be considered the reference position and shall be the position that all samples will be mounted when a roughness measurement is made. A two axis motorized stage shall be used to position the stylus of the roughness profilometer over the desired positions of the sample. A third motor shall be used to lift and lower the stylus onto the sample such that no manual manipulation of the device is required. The control and test regions of the specimen shall be divided as shown in Figure 1. Five readings shall be taken on the control region, and five readings shall be taken on the test region of the specimen. The measurements shall be taken 3 mm from the centerline dividing the control and test regions of the specimen as shown in Figure 1 using the two axis motorized stage to ensure precise positioning. Furthermore, each group of five measurements shall be equally spaced 1.5 mm apart as shown in Figure 1.
5. **Polishing of specimen.** After measuring the height, gloss and surface roughness of the specimen, it shall be polished using the respective polishing system to be tested.
    a. The specimen shall be mounted in a custom-built polishing apparatus, as shown in Figures 2 and 3.
    b. The speed of the handpiece shall be set at 17,500 rpm (research by Jones *et al*. 2005 showed that this is an optimum handpiece speed for polishing composite resins), unless a specific speed is specified by the manufacturer of the polishing system for optimum performance and then that speed shall be used. If a range is given by the manufacturer, the maximum speed shall be used.
    c. The polishing system shall be mounted at a 10 degree angle from the plane of the face of the specimen unless a higher angle is required

(shown through pilot studies) in which case the lowest angle that will be allowed by the polishing system shall be used.

d. The specimen shall be mounted to a vertical slide that is attached to a computer-controlled motor, which can move the specimen into the polishing device until a programmed load is attained. The load that the specimen contacts the polishing device with shall be 30 g (research by Jones *et al*. 2005 showed that 20-30 g is an optimum load for polishing composite resins).
e. Once the specimen contacts the polishing device with 30 grams of force, the motorized positioning table shall move the specimen unitdirectionally the full distance of the diameter of the specimen beneath the polishing device for 10 seconds while the computer-controlled motor maintains the 30 g load. After 10 seconds, the specimen shall be removed from the polishing device, rinsed with a stream of deionized water for five seconds, and dried with a stream of filtered, compressed air for five seconds.
f. The surface roughness of the sample shall then be recorded as described in step 4 above.
g. The gloss measurement of the sample shall then be recorded as described in step 3 above.
h. The specimen shall then be mounted back in the polishing apparatus. Note: Pilot studies were performed to verify the useful life of each step of the multi step polishing systems. The number of passes required before switching to the next step was based off of these pilot tests. For multiple step polishing systems, after reviewing the average roughness data verify that the roughness measurements have plateaued with the current polishing disk. After this has happened the next polishing device shall be used to polish the sample for 10 seconds as described above. For one-step polishing systems, the same polishing device shall be used to polish the sample for 10 seconds as described above. For polishing systems that have a manufacturer note as to a contact time limit, the polishing device shall be replaced after that time limit has been attained.
i. After 10 seconds, the specimen shall be removed from the polishing device, rinsed with a stream of deionized water for five seconds, and dried with a stream of filtered, compressed air for five seconds.
j. The surface roughness of the sample shall then be recorded as described in step 3 above.
k. The process of polishing the sample for 10 seconds followed by rinsing, drying, measuring the surface roughness and gloss of the specimen shall continue with the most recent polishing apparatus until the average recorded surface roughness is less than 0.2 μm, which according to a clinical study on implants is the critical threshold value for bacterial retention ([Quirynen *et al.*], it has also been reported that restorations must have a maximum surface roughness of less than 0.5 μm so as not to be differentiated from the surrounding enamel by a patient's tongue [Jones *et al.* 2004]) or until the specimen has been polished 12 times. NOTE: During each of the polishing steps, the surface roughness of the control region shall be monitored to make sure that the readings are the same within the precision of the surface profilometer. If there is a significant deviation from acceptable values, the cause of the deviation

shall be determined and corrected. If it is a problem with the sample, it shall be discarded and a new sample shall be made and tested.

l. After all 12 passes have been completed the specimen shall be mounted back on the mechanical breadboard and the heights of the same points as described in step 2 shall be checked and the change in heights recorded.

6. **SEM pictures**. SEM micrographs shall be taken of the surfaces of the polished and control regions of the specimens so that representative pictures can be shown in the issue of the *ADA PPR*. Likewise, SEM micrographs shall be taken of the polishing devices before and after they are used to polish the specimens.
7. **Results**. For each polishing system manufacturer, results shall be reported for each type of composite material. This shall include average surface roughness values at each time interval along with the average total change in height, and the average total time for all of the polishing steps. Furthermore, the average gloss values at each time interval shall be reported.

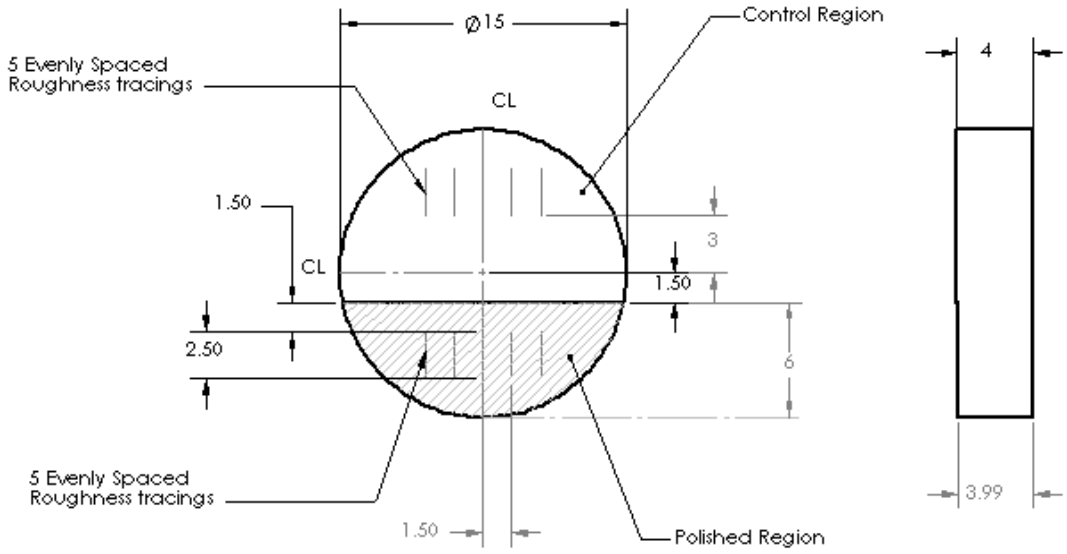


**Figure 1:** Top and side view of sample specimen showing the control region and polished region along with the locations of the surface roughness measurements. Dimensions are in millimeters.

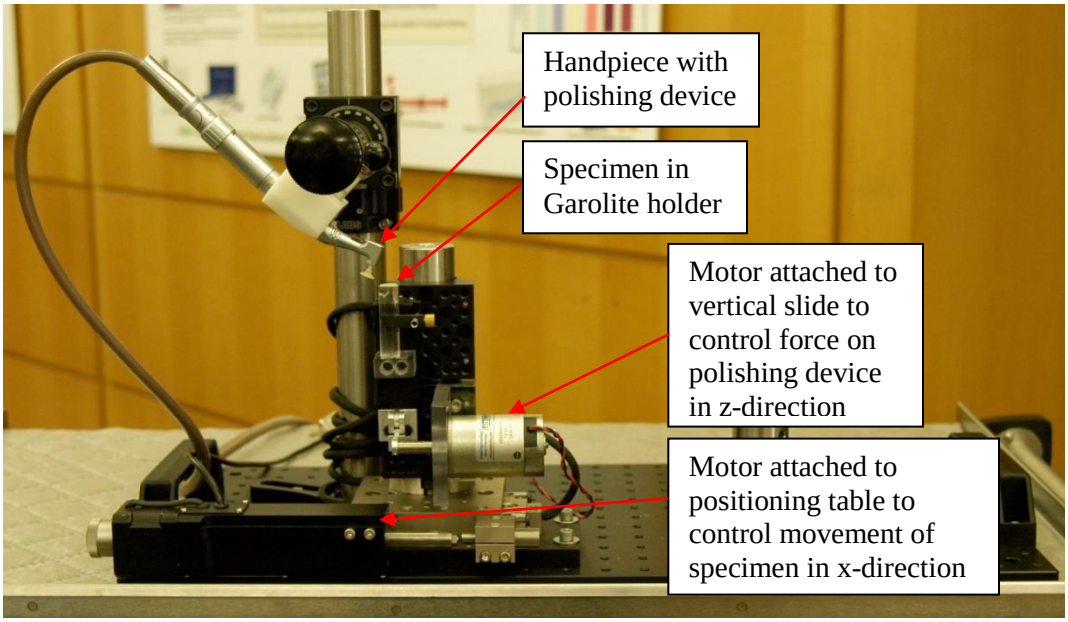


**Figure 2:** Picture of custom-built polishing apparatus.

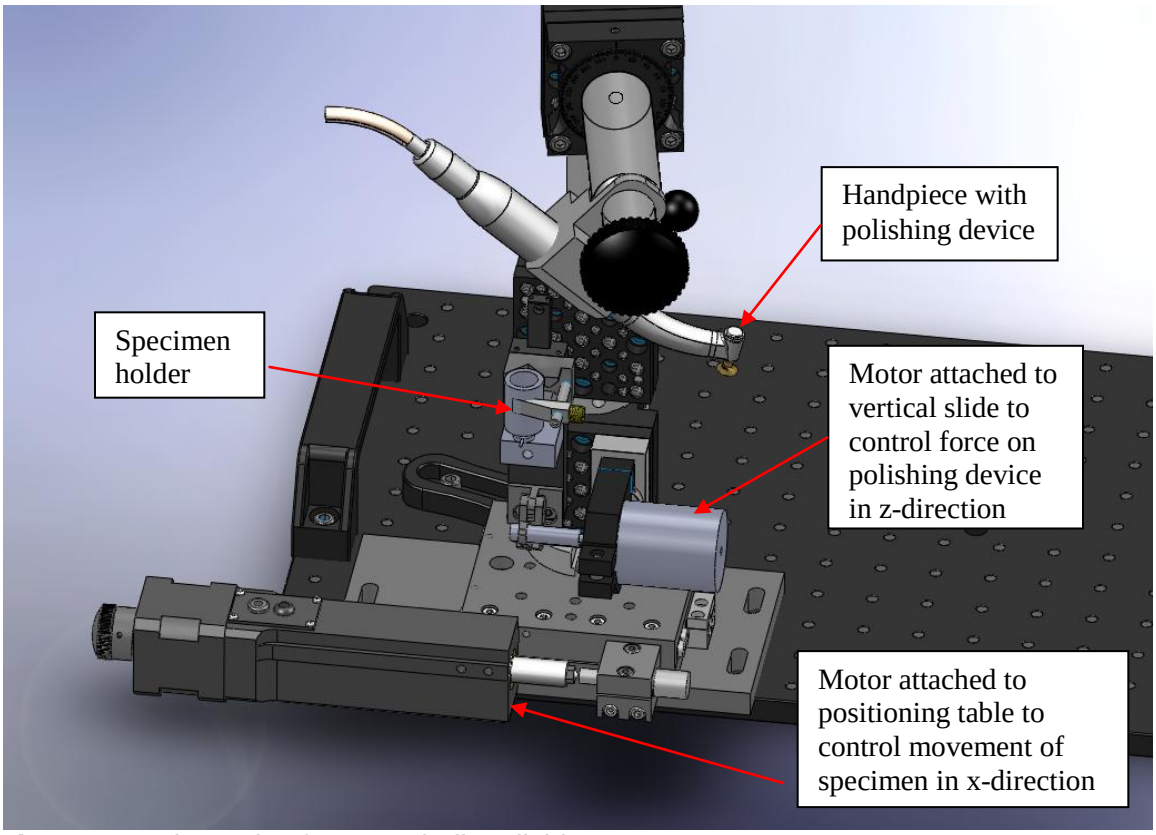


**Figure 3:** Schematic of custom-built polishing apparatus.