Evaluating subsurface damage in optical glasses

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Hard brittle materials (e.g. glasses and ceramics) increasingly appeal to general interests because of their excellent physical, mechanical and chemical properties such as super hardness and strength at extreme temperature and chemical stability. The precision manufacturing of these materials is primarily achieved by grinding and polishing, which generally employs abrasives to wear the materials. With this manufacturing technology, the materials are removed due principally to the fracture of brittle materials, which will leave a cracked layer on the surface of manufactured components, namely subsurface damage (SSD). The subsurface damage affects the strength, performance and lifetime of components. As a result, investigation into the subsurface damage is needed. A host of characterizing techniques have been developed during the past several decades. These techniques based on different mechanisms provide researchers with invaluable information on the subsurface damage in various materials. In this article the typical SSD evaluation techniques are reviewed, which are regularly used in optical workshops or laboratories. [DOI: 10.2971/jeos.2011.11001]

Keywords: fused silica, grinding and polishing, optical manufacturing, destructive evaluation, non-destructive evaluation

1 INTRODUCTION

The relatively larger-abrasive grinding of brittle materials usually causes a cracked layer on the top layer of optical glasses, namely subsurface damage (SSD) which usually takes the form of microcracks. Subsurface damage resulting from the mechanism of brittle material removal can weaken the strength of material, serve as a reservoir for laser light absorbing precursors and polishing contaminators, enhance the electric field inside the cracks, and thus greatly influence and affect the operational durability and lifetime of component in high power laser systems, semiconductor industry, military and astronomical applications [1]-[8]. In typical optical manufacturing processes, subsurface damage is introduced during the first stages of cold processing (mostly in grinding processes) and diminishes in subsequent processes [4]-[11]. Material is removed with progressively finer abrasives and each step removes sufficient material on the surface of substrates in order to get rid of the subsurface damage layer left by previous steps and to eventually reduce subsurface damage as much as possible [4]. To suppress/eliminate subsurface damage and obtain perfect manufactured surface is the ultimate goal of optical fabrication. Considerable efforts have been made to achieve the goal and some newly proposed technologies (e.g. deterministic microgrinding, ductile grinding of brittle materials, elastic emission machining, magnetorheological finishing, reactive atom plasma (RAP) processing etc.) show great potentials to shorten the whole processing time and/or give rise to little damage to the surface of optical substrates being processed owing to their unique mechanisms of material removal [12]-[20]. But at the most optical workshops optics are finished by skilled opticians with conventional manufacturing technologies that do not remove/eliminate subsurface damage completely. On the other hand, subsurface damage has been proven to strongly depend on the manufacturing conditions [4], [10], [21]-[27]. Thereby, evaluating subsurface damage in ground/polished optical parts plays a pivotal role in optimizing manufacturing processes to improve processing efficiency and to reduce time and cost.

A number of methods have been applied to evaluate subsurface damage, which significantly promote optical fabrication [28]-[32]. Speaking generally, these methods fall into two categories: destructive and non-destructive evaluation. The destructive methods can measure subsurface damage pre-

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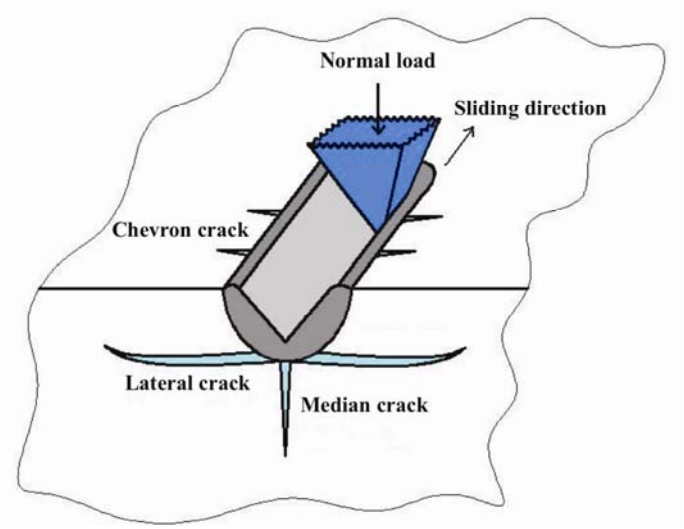


FIG. 1 The mechanism of material removal of brittle materials in brittle mode: microcracks emanate from the boundary of plastically deformed region which is immediately beneath the indenter; when the lateral cracks intersect the surface of brittle material, the material is removed as chips; the median cracks can extend much farther below the surface and thereby form subsurface damage [adapted from reference 35].

cisely and quantitatively and can provide useful information on the specimens being tested, but they may be time -consuming and inevitably alter or even destroy the finished surface of the samples. Consequently, the samples may be unusable any more and the cost of production may be raised. Another drawback to destructive methods is that the methods are generally statically meaningful and it is unlikely to inspect every sub-area of the finished optics. Thus the subsurface damage of measured subarea may not fully reflect the characters of the whole sample. Therefore, some non-destructive means were put forward to examine optical components quickly without damaging samples. Many practical methods were introduced over the last several decades; these techniques work well for specific materials and fabrication processes. Nonetheless, the non-destructive measurements have obvious limitations: they are generally quite operator dependent; they sometimes provide only qualitative data; they require the specimen must be testable; the apparatuses for nondestructive measurements are usually expensive and the mechanisms are rather complicated; etc. [32].

In this review, we summarize some representative techniques used to evaluate subsurface damage in optical glasses, especially fused silica, one of the most important engineering materials. In Section 2, how subsurface damage originates during the machining of optical materials is presented and the applicability and limitation of each technique are analyzed in Section 3 and 4. We hope, to the best of our ability, to provide scientists and engineers concerned with subsurface damage in optical manufacturing with a corpus of prevailing SSD detecting methods as well as emerging techniques.

2 SUBSURFACE DAMAGE IN BRITTLE MATERIALS

Thanks to their excellent physical and mechanical properties, such brittle materials as glass, ceramics and glass-ceramics are increasingly attractive in many engineering applications. However, due to high hardness H (resistance to plastic deformation) and brittleness (which can be simply defined as H/Kc, where Kc is fracture toughness, resistance to fracture [33]), these materials are difficult to be machined. In general, the grinding and/or lapping are employed to precisely machine these materials in optical manufacturing and this manufacturing process usually generates sub-/surface damage in the surface of brittle materials as a result of brittle mechanism of material removal (Figure 1) [9], [34]-[38].

The ground surface is transferred to polishing workshops where the optics is polished to specular surface with proper abrasive and polishing laps, the subsurface damage is removed and the surface form error is further corrected [39]. The sufficient material should be removed in the polishing process so as to eliminate the SSD left by grinding/lapping [4]. However, polishing is a complicated chemical-mechanical process during which a hydrated layer is given rise to and deposits on the top surface [40], [41]. It is this thin layer that contributes to the removal of glass in glass polishing process [40], but this layer is a double-edged sword and it is also this layer that covers and conceals the damage originated in grinding and polishing processes which should be polished out completely. Therefore, more often than not, in spite of no damages or defects on the surface of the optics, the surface surprisingly presents quite a few digs, pits, scratches, etc. after chemical etching [42]. Accordingly, the subsurface damage needs evaluating before/after the polishing process in order to ascertain whether the subsurface damage is controlled below the desired level. It is noteworthy that the scratches/pits under the hydrated layer after polishing are referred to as UNUSUAL subsurface damage since the residual layer masks the scratches/pits and makes the scratches/pits appear to be SUB-surface damage [43]; it is the interpenetrating cracks in the top layer of the ground surface that we call subsurface damage [43]. The SSD in this review refers to microcracks rather than UNUSUAL surface damage (i.e. scratches, digs, etc.) unless otherwise specified. The conceptual illustration of subsurface damage during manufacturing an optical component is shown in Figure 2 [6], [44], [45]. It is worth mentioning that no distinct boundary exists and there may be some transitional areas between each two layers in Figure 2(b).

The research on subsurface damage of optical components dates back to early 20th century when Rayleigh and Preston used hydrofluoric acid to erode glass [46], [47]. Preston etched fine ground glass and found the etched surface was full of valleys and digs which, the author believed, originated in flaws left by grinding operation; these digs and valleys now are referred to as subsurface damage. This meth-

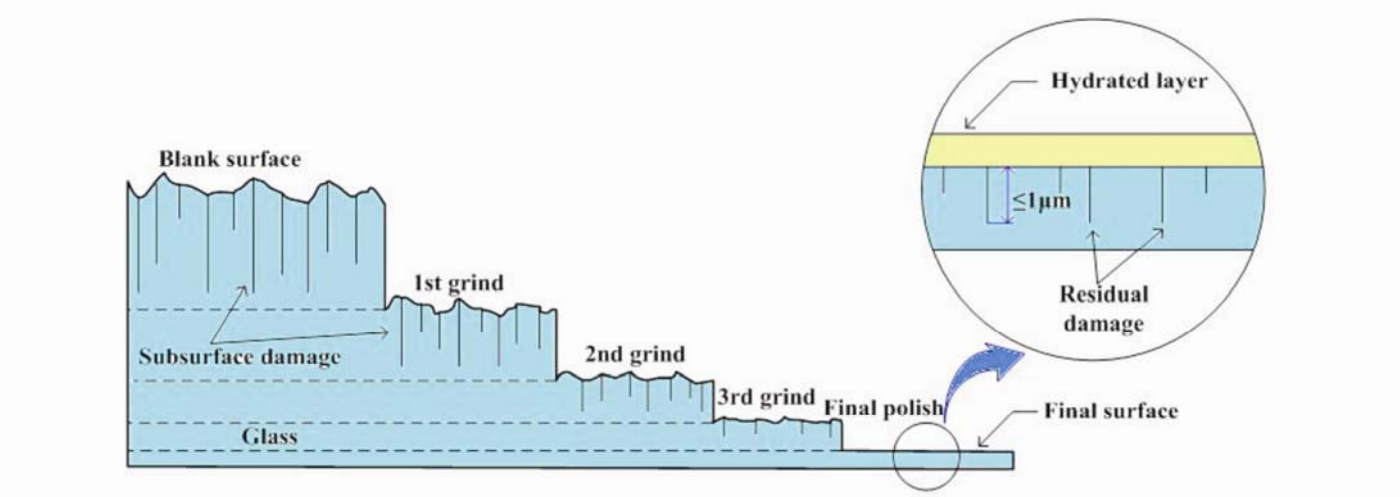


FIG. 2a) The schematic view of successive steps from shaping to polishing of an optical component; each grinding step generates its own characteristic distribution of subsurface cracks while polishing gives rise to a hydrated layer depositing on the surface [4].

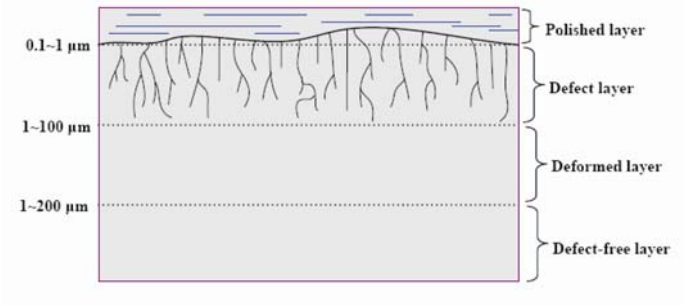


FIG. 2b) The schematic illustration of subsurface damage. The top surface is a thin layer of hydration which is introduced during the polishing. Next is the microcracks left by grinding/lapping process. Then the deformed layer follows which also is initiated in the grinding/lapping. All of these first three layers are extrinsic and undesirable, beneath which is the damage-free bulk material [44, 45].

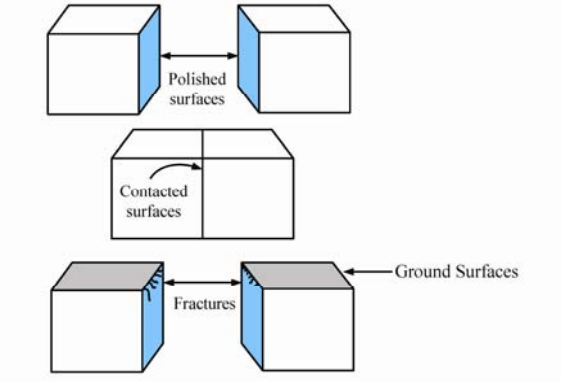


FIG. 3 Sketch of the Bonded-Interface Technique: Two identical blocks integrate with each other through optical adhesive; the contacted surfaces are polished in advance, respectively. Then the integrated top surface of the combined block is ground, indented, or scratched along the direction not parallel to the glue layer. Thereafter, the glue is chemically resolved and the subsurface damage can be inspected conveniently with microscopy on the polished surface of any independent block [54]

od is still widely used as a precise inspection technique for testing SSD until now. Since chemical etching unavoidably alter/change the surface of optics, some nondestructive methods were proposed over past tens of years. The nondestructive techniques are based on the fact that the properties of the detecting light, acoustic wave, electromagnetic wave will change once encountering the subsurface cracks when they penetrate into the tested materials [48]. The destructive and nondestructive techniques for evaluating SSD will be introduced in the following two sections, respectively. Undoubtedly, the classification is not absolute and there can be many classifications according to different criterions. For instance, chemical etching method as well as dye impregnation is also viewed as nondestructive techniques by other researchers.

3 DESTRUCTIVE EVALUATION

Destructive methods involve physical modification in components to expose the structure below the ground surface. It is common to use polishing to obtain the morphology of subsurface, because polishing will in principle induce little additional damage to materials. Another kind of method is chemical erosion that removes material without bringing about new fractures. These techniques generally need microscopy to facilitate measurements, which are usually time-consuming and tedious, though they are much reliable and accurate. Some means will be discussed below.

3.1 THE BONDED INTERFACE TECHNIQUE

The bonded-interface approach was first used by Mulhearn and others to examine the SSD in ground optics and the SSD resulting from indentation or scratching [49]-[54]. Two square samples with identical shape and material are glued together using adhesive to form a combined one, which is preceded by the polishing of the surfaces to be touched face to face (Figure 3). The combined block is ground or scratched to generate subsurface damage. Following that, the adhesive/glue is resolved chemically, which should not damage the surface or affect the subsurface damage of optics. Alterna-

tively, the prepared sample can be halved with extreme caution after being ground. Thereafter, the sample is chemically etched for the purpose of opening the optically contacting microcracks. Then the subsurface damage of ground optics is apparent on the lateral surfaces and ready for inspection. The subsurface damage can be examined with an optical microscope or scanning electron microscopy, etc.

This technique provides a direct observation of SSD compared to other destructive methods, but the surface should be carefully polished/ground or halved in order to prevent additional damage from intervening the measurement of the desired subsurface damage. The method, in fact, is slightly different from grinding process. The SSD in an intact sample can be viewed as to occur in infinite space, while the SSD in bonded interface technique is generated in half infinite space. However, this difference does not alter the characteristics of SSD in samples [54].

3.2 TAPER POLISHING, MAGNETORHEOLOGICAL FINISHING (MRF) WEDGE, BALL DIMPLING AND MRF SPOTTING

These techniques are substantially the same, which are based on the fact that polishing introduces no new observable subsurface damage [6], [21], [23], [24], [55]-[59]. The polishing processes used in taper polishing and dimpling were put forward several decades ago [46], [60]-[62], but they are now superseded by the MRF because the MRF possessed advantages over traditional polishing techniques in terms of subsurface damage. In the magnetorheological finishing (MRF), a magnetic-field-stiffened ribbon of fluid is applied to polish out an optics [14]-[16]. As a state-of-the-art polishing technology, the mechanism of material removal of MRF differs from the traditional pitch/pad polishing. In MRF, the material removal is due to the great tangential effects as opposed to normal force in conventional polishing [37], [63]-[65]. Shorey [64], [65] and Miao [63] measured the material removal rate with varieties of polishing abrasives and the shear stress in MRF polishing to confirm that the shear effect plays an essential role in material removal and the shear stress is determined by mechanical properties of materials in MRF [66]. It is the mechanism of the tangential effects in MRF that significantly eliminate and almost do not initiate subsurface damage when finishing an optics. Accordingly, the MRF is gradually adopted as an effective tool to expose the material below the surface. The deepest polishing region under ground surface ought to exceed the depth of SSD and extend into the bulk material.

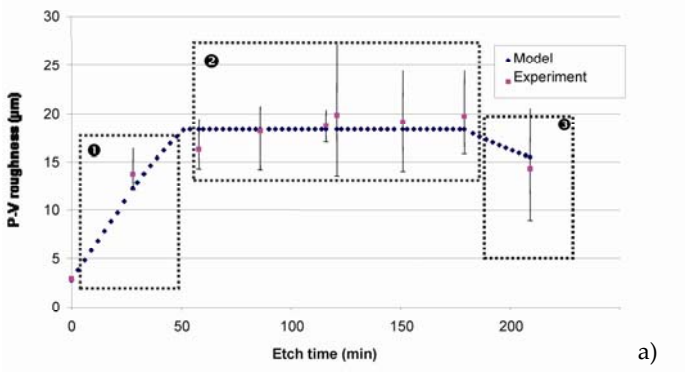
With the ease of examination under microscope, the processed sample is chemically etched so that the microcracks are opened and enlarged. The chemical solution is usually made up of aqueous HF and additive of NH_4F or strong acid (HCl, HNO_3, or H_2SO_4, etc.) and occasionally the heated strong alkaline NaOH or KOH is employed. The aim of adding NH_4F is to stabilize the etching rate of fused silica, while the addition of strong acid can accelerate the etching rate markedly [67].

These methods are widely employed in optical fabrication community at present due to cost-effectiveness, reliability and simplicity. The methods generally involve an optical microscope and a contact stylus profilometer. The polished spot/wedge is first profiled with the profilometer along the centerline of spot or the wedge; afterwards, the sample is placed onto a platform under the microscope. Scanning the spot/wedge with the microscope along the same path as the profilometer and then comparing the results of microscope with those of the profilometer, we will acquire the depth of SSD and the morphology of SSD at varied depths. The optical microscope can be replaced with an atomic force microscope (AFM) in this procedure [68]. More recently, a modified method was used to simultaneously obtain the depth of SSD and morphology of SSD at varied depths without the need for prohibitive profiler [69]. This method is based on the small depth of field of high numerical aperture (NA) to precisely resolve the depth of SSD. The incorporation of a laser displacement sensor facilitates the non-contact SSD measurement at different depths.

3.3 CHEMICAL ETCHING METHOD

The recently developed chemical etching method measures the variations in surface roughness of ground fused silica with the etching time or material etched away. According to the chemical corrosion, the fused silica will be isotropically attacked by chemical molecules on the interface between the solid fused silica and the solution; therefore, the profiles of the subsurface microcracks will be enlarged literally [70]. Then the enlarged cracks are readily tested with suitable contact stylus or optical profilometer [7]-[8], [71]. The measured surface roughness increases initially and reaches a plateau with the etching, followed by a smoothly decreasing with protracted chemical etching time (Figure 4(a)) [7]-[8], [71]. The surface roughness in the plateau is viewed as subsurface damage. The etching method is also employed to reveal scratches or digs on polished optics (i.e. unusual subsurface damage), which are mantled by the re-deposition layer resulting from the interaction between glass, aqueous slurry and polishing tool [45], [72].

As a matter of fact, the measured surface roughness should keep almost constant and tend to trail off beyond a critical time/thickness. The surface roughness of ground isotropic optics behaves like a step function in this process. Nevertheless, if one measures the surface roughness with a contact stylus profilometer, it is very likely to find that the surface roughness increases at first, then tends towards stability and drops. That is due to the fact that the radius of stylus tip is not infinitesimal [73]-[75], which will result in the plausibly small measurement of surface roughness at the initial stages (Figure 4(b)). As a consequence, the results take the form of a trapezoid. Theoretically speaking, an ideal stylus tip will yield results that the surface roughness will not increase at the first stage of etching and will decrease beyond a critical thickness [70].



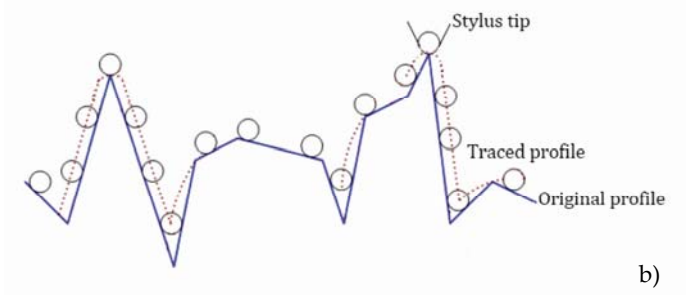


FIG. 4 The surface roughness with the chemical etching. (a) The peak-to-valley surface roughness (ground fused silica sample): the surface roughness increase in the beginning; after several minutes, the surface roughness is stable; at last the surface roughness exhibits the sign of decreasing [7]. (b) The actual surface profile and the measured profile with a contact stylus. The measured profile is distorted due to finite dimensions of the tip of the contact stylus profilometer [75].

It is also practicable to check the constancy of etching rate versus time or thickness of etched material to determine the subsurface damage. The ground optics definitely contains countless microcracks and stress at the tip of microcracks. When ground isotropic optics is subjected to chemical etching, the strained and cracks-containing layer will be dissolved faster than bulk material and cracks are increasing enlarged which is accompanied by stress releasing [70]-[76]. The actual contact diminishes between the etchant and glass after a critical time/thickness. At last, the rate will level off. The etched thickness at which the rate tends to steady can be referred to the SSD depth.

In spite of the convenience of chemical method due to the absence of polishing, chemical etching methods apply to only isotropic materials because the etching rate and resultant surface roughness of anisotropic materials are not only influenced by SSD but also other factors. Accordingly, etching rate and surface roughness will not indicate the SSD reliably.

3.4 Focused Ion Milling (FIB) cross sectioning, MRF 3D cross sectioning

The FIB cross sectioning was used to reveal the cracks under scratches or indentations [77]-[86]. With regard to the detailed development of FIB, the paper by Sugiyama & Sigesato is recommended [87]. This technique was first applied to dislocations, phase and structural changes of ceramics and metals, and later extended to cracks induced by indentation in soda-lime-silica glass [88]. The FIB incurs difficulty in observing a wide section due to the fact that FIB only exposes the fairly limited area of concern to observers. The 3D FIB involves the reconstruction of 2D images that a serial 2D tomography is created with a highly focused ion beam and imaged with ion-induced secondary electrons (ISE) and then reconstructed with professional software [89]. The 3D FIB is capable of quantifying the size of cracks at high spatial resolution up to <100nm, which makes it an ideal site-specific analyzing and nano-processing technology. However, the FIB technology has been notorious for material re-deposition effects when cross-sectioning the samples. The adverse effect can be relaxed through adopting an inclined incident ion beam. Like 3D FIB, the magnetorheological finishing (MRF) was recently applied to generate 3D cross-sectioning because of its unique material removal mechanism that little/no sub-/surface damage is incurred [90]. These two methods both need repetitive polishing; moreover the MRF 3D also requires precise re-positioning of the sample. The accurate 3D reconstruction of these two methods is dependent on the incremental between sequential 2D slices, since the regions between two slices are approximately interpolated. In addition, it should be pointed out that the high energy FIB sputtering brings about changes in residual stress and structure and therefore damaged artifacts in the samples, which might alters the crack distribution of the samples that are FIB -processed. To date there is no satisfactory solutions to the problem.

3.5 Dye impregnation

Because the ground optical parts usually contain a large number of microcracks in the surface layer, the dyes contrasting in color against optical substrate or contrasting against substrate after being irradiated with X-ray, laser, etc. can be pressed into the materials [91]-[95]. If the substrate is composed of dark color materials, the fluorescent dyes can be utilized. Then the subsurface damage is readily inspected optically or electronically. Since the cracks, in effect, optically contact, the penetration depth where dyes permeate is not sufficient and may not represent the real depth of microcracks. In order to obtain the crack depth as precise as possible, some methods for impregnating the dyes have been proposed: mechanical preloading [91] and cold isostatic pressing [93] and electron probe microanalysis (EPMA) was employed to probe the trace of the dye in extreme small concentration (Figure 5). The tensile stress is exerted on the substrate to open the microcracks due to bending in the preloading. The stress is adjusted so that it does not exceed the half of the mean failure stress of substrate and resultant damage to substrate. Following the loading, the load is unloaded im-

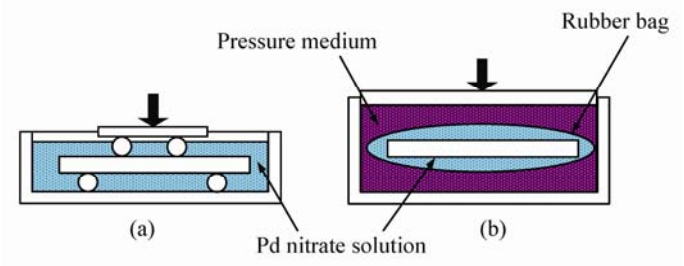


FIG. 5 Two dye impregnation methods: (a) preloading method and (b) cold isostatic pressing method. Upon impregnating the dye into the samples, the samples can be observed using ordinary optical microscope to acquire the knowledge of subsurface damage under the illumination of laser light, X-ray, etc. [93].

mediately without any delay. One may question whether the method suffers from the propagation of the cracks during the loading/unloading processes. Choi and Salem [96] have shown that the preloading up to 90% of failure stress has little influence on fatigue parameter (n) for most brittle materials like glass and ceramics, and therefore the crack propagation owing to preloading can be neglected. The cold isostatic pressing forces the dye to impregnate the microcracks by use of fluid pressure medium. The air contained in the cracks will dissipated by high pressure dye fluid (e.g. Pd $(NO_3)_2$ solution). The technique is especially favorable to shallow open cracks in the surface of ground optics. Once it is mapped by electron microprobes, the distribution of palladium will reflect the morphology of the crack.

4 NON-DESTRUCTIVE EVALUATION

Although they are quite accurate, the destructive evaluations, in general, are time consuming and labor intensive; in addition, they inspect only localized areas and unavoidably destroy the substrate tested. As a consequence, a variety of methods are developed to evaluate SSD nondestructively and speedily and therefore cost-effectively. The techniques to be described are representative of the numerous nondestructive evaluations (NDE) to detect the SSD in glasses.

4.1 Estimation of SSD from the surface roughness and abrasive size

Preston [47] originally observed that a great number of flaws left by grinding operation extended to a depth two or three times as great as that of the deepest pits; Kachalov [97] reported that the depth of the damaged layer was proportional to the peak-to-valley surface roughness of ground surface, who first related the subsurface damage to the surface roughness of ground glass and developed by other researchers [6], [8], [10], [12], [21], [37], [57]-[59], [98]-[103]. In grinding/lapping of brittle materials where large abrasives are used, the abrasive particle acts as a loaded indenter which slides/rolls on the surface of optical substrate (Figure 1). The surface underneath the indenter will fracture because of loading and unloading on the indenter when the load exceeds a certain value. The depth of median cracks can be viewed as the subsurface damage while the lateral cracks will constitute the surface roughness after grinding of the brittle materials. As a consequence, there exists a relationship between the subsurface damage and surface roughness (Table 1). Thereby we can estimate the ranges of the subsurface damage of some materials under certain conditions once the surface roughness is known. Because the surface roughness can be described by many parameters (R*t*, Rz, R*a*, R*q* etc.) [104], the relationship will vary from one parameter to another. Most researchers linearly relate subsurface roughness to peak-to-valley roughness, while Li et al. linked subsurface damage with Rz roughness who believed a nonlinear relation between SSD with surface roughness [57]. Nevertheless, other researchers argue that the peak-to-valley surface roughness is preferable to root-mean-square (RMS or R*q*) or average (R*a*) surface roughness [22]. Lambropoulos et al. have shown that the depth of SSD is not more than twice the peak-to-valley roughness of a well ground surface [37], [56], [105]. However, the surface quality may not be always a reliable indicator of SSD in some cases: cracks may extend to a much deeper levels below a flatter and smoother ground surface than a surface ground more roughly [106]. In addition, the surface roughness is greatly influenced by the measuring apparatus (Figure 4(b)) [73]-[75]; hence the proportional coefficient varies from laboratories to laboratories.

	Ratio of SSD to surface roughness (R*t*)	**Materials**
Preston [98]	3~4	Glass
Aleinikov [56]	3.9±0.2	Glass, marble, crystal and ruby
Miller et al. [21]	9.1	Fused silica
Kachalov [97]	3.7~4	Glass
Hed and Edwards [100]	6.4±1.3	BK7, fused silica and Zerodur
Randi et al. [56]	1.4	BK7, fused silica, Si, BSL7, $LiNO_3$, CaF_2, MgF_2
Neauport et al. [8], [103]	~9 3.3±0.5	Fused silica (for diamond grinding) Fused silica (for loose abrasive grinding)

TABLE 1 Subsurface damage can be related to surface roughness when glass and ceramics are ground in brittle removal mode. The proportionalities of subsurface damage to surface roughness by several scientists and engineers are summarized. The surface roughness refers to the peak-to-valley (R*t*) roughness of the substrate.

Another estimation of SSD comes from the abrasives used in grinding/lapping. Based on numerous experiments in 1930s~1950s, Kachalov [97] gave a simple linear relationship between SSD and the maximum size of abrasives used. Sabia [107] stated in his/her paper that the SSD is proportionate to 5 times the mean diameter of the abrasives for fixed-abrasive grinding; the proportionality ranges from 1~1.8 times the mean size of used abrasives in loose abrasive grinding/lapping. Ma et al. [108] tested the SSD with confocal scattering microscopy and found that the depth of the SSD was 4~6 times the size of abrasives. After experimenting on many glass and ceramics under miscellaneous conditions, Lambropoulos has narrowed the range of the depth of SSD [109]:

$$0.3d^{0.68}<\mathrm{SSD}<2d^{0.85}$$

where, the abrasive size d and the depth of SSD are in μm. Our recent experimental results that the ratio of SSD depth to the nominal abrasive size lies within the ranges of 0.2~0.5 and 0.4~1.6 for bound-abrasive and loose abrasive grinding, respectively, corroborate the conclusion [110], [111].

In recent years, Suratwala et al. [21], [22], [112] have established a correlation between the maximum depth of SSD and the average crack length which can serve as helpful guidance to estimate the SSD and therefore to optimize the manufacturing processes. The estimation of SSD from either surface roughness and abrasive size or the crack length and crack wide considerably rely on the measurement of these factors that will vary among the different researchers and instrumentation, though the method may be the simplest and most cost-saving in optical shops.

4.2 Laser scattering and confocal microscopy

The laser scattering, one of nondestructive characterization methods, is at first studied for surfaces quality and then extended to subsurface characterization [113]-[118]. When a beam of light penetrates into a transparent or translucent surface, the optical scattering will take place on both the surface and subsurface owing to non-ideal smooth surface and subsurface defects. The reflected light, transmitted light, scattered light and subsurface scattered light appears together. Therefore, it is essential to distinguish subsurface scattered light from scattered/reflected surface light in a subsurface characterization system. To this end, a polarized laser and a polarization analyzer are adopted, which is referred to as cross-polarization technique. In a scattering system, a detector is placed in order to detect and record the scattering of subsurface damage. Then the depth of surface/subsurface defects can be roughly determined by the intensity of the scattering and the change in polarization angle. However, this technique cannot provide precise knowledge of the depth of defects since the detector receives all the measurable scattered light. But this problem can be overcome by incorporating the confocal microscope into the laser scattering system. Sun et al. [119], [120] combined the cross-polarization laser scattering and confocal microscopy to detect the subsurface damage of transparent and dense materials with depth resolution of <1 μm [121]. Lu et al. used a similar setup, cross-polarization confocal microscopy, to measure SSD of ground silicon wafers [115]. The usage of a polarized laser (He-Ne laser: @ 633 nm) and a polarizing beam splitter (PBS) differentiates this system from an otherwise identical conventional confocal microscopy. All light scattered from surface will not change the polarization while that scattered from subsurface will be reflected and refracted and therefore depolarized due to discontinuities like microcracks. The light scattered from subsurface will be directed towards the recording detector preceded by a stainless steel pinhole and imaging lens. Due to the fact that rough surface may alter the polarization of scattered light, this system is not well suited to the substrate with quite rough surface. In addition, it is required that the material to be tested be transparent or translucent with respect to the incident light.

The confocal microscopy is involved in the above method. The confocal microscopy was originally used to view samples in biology [122]-[123]. Not until recent years is this advanced technique applied to technological materials such as glasses and ceramics and to the measurement of the line width and overlays in semiconductor manufacturing [108], [123]-[134]. Optical slicing and three-dimensional reconstruction of materials can be realized with the technique because the light reflected or scattered out of the focal plane is blocked in the optical system. Confocal scanning laser microscopy can be used to acquire subsurface information of opaque ceramic materials [135]. The top 20 μm subsurface structure beneath the surface can be obtained for some ceramics. Neauport et al. [136] have discussed the application of confocal fluorescence microscopy to observation of the subsurface damage of ground optical substrate. This technique needs surface pre-treatment of the opposite surface of the substrate in order to image the subsurface damage clearly when the roughness of surface to be observed is very high. The image will be blurred with increasing the surface roughness until the microscope is unable to yield details of subsurface damage. Comparatively speaking, the technique is complicated and time consuming, and sometimes, for example, very coarse surface, the depth of SSD cannot even be acquired. Derndarsky and Oklind [137] have likewise demonstrated that the subsurface damage of use-wear in quartz can be apparently displayed under the fluorescent mode of the confocal laser scanning microscope in combination with dye to intensify the fluorescent light so as to make cracks beneath surface visible and detectable. Fine et al. [138], [139] have reported a recently improved laser scanning confocal microscope that can directly and conveniently inspect the SSD of optics either polished or finely ground (Figure 6). The confocal scanning laser microscope is adopted in surface-scanning mode, but the focal plane is located inside the optical substrate instead of the surface. When scanning the substrate vertically (Z-direction), the 3D profile of the optical substrate will be created. Moreover, the resolution of 150 nm has been reached [138], which is related to the microscope parameters, such as numerical aperture, the wavelength used, etc. This

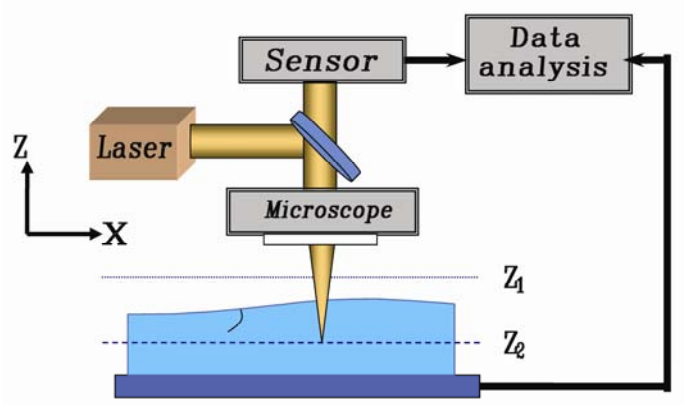


FIG. 6 The principle of confocal microscopy developed by Agilent. The substrate should be transparent or translucent with respect to the illuminating laser light. This apparatus is not applicable to very low quality of surface (e.g. coarse-abrasive-ground surface) [138].

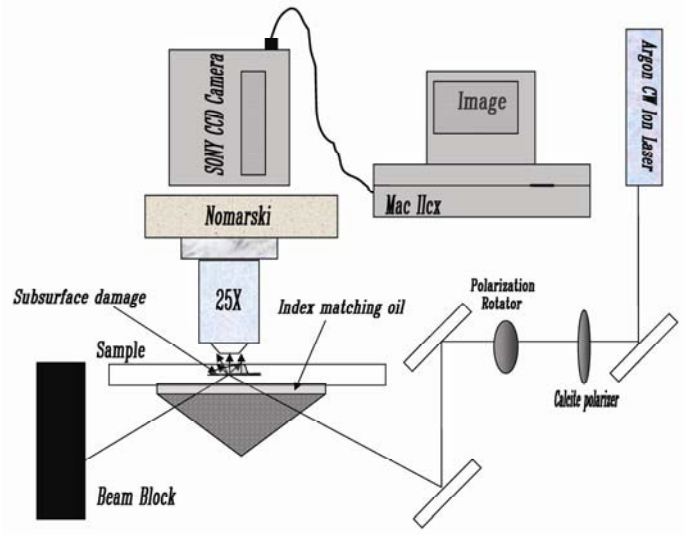


FIG. 7 The configuration of the total internal reflection microscopy set up at LLNL. The sample surface should be smooth enough so that the incident laser light can penetrate into the bulk and be reflected out of the sample when the requirements for total internal reflection are satisfied [147].

technique is fairly convenient, thought it suffers from the surface requirements like Neauport's method [136], that is, when the substrate possesses disappointing rough surface, the measurement system is incapable of testing the SSD. Additionally, this testing system convert the apparent depth to the actual depth of SSD due to refractive index of the substrate, which also incur some troubles determining the SSD because the top layer full of cracks may differ from the bulk material in the index of refraction [140]. Confocal microscopy has also been employed to investigate the damage mechanisms in fused silica optics when subjected to intense laser pulses [141]. The surface and subsurface morphologies beneath the damage spots is explored with a confocal microscopy with resolution of 1 micron and 0.26 microns in vertical and transverse directions, respectively.

4.3 Total internal reflection microscopy (TIRM)

Total internal reflection microscopy is another non-destructive inspection method for surface and near surface damage, which shows the potential to assess polishing residual damage, inclusions of coated optics, and scattering sites probably leading to laser damage of optical component [142] -[149]. Strictly speaking, TIRM belongs to laser scattering techniques, too. The TIRM setup originally conceived by Temple [142], [143] and developed by other researchers [144] -[148] is shown in Figure 7, where a laser beam is linearly polarized after passing through a polarizer and then the desired S-polarized light is singled out owing to stronger scattering than P-polarized light. Then the laser beam steered by mirrors and lenses is incident onto the surface to be tested by passing through bottom or lateral surface of the sample (Figure 7) at an angle that satisfies the requirements for occurrence of total internal reflection. When there are no defects within the sample or on the sample surface, the laser light will be reflected totally by the air-glass interface. However, the laser light will be scattered in the presence of defects and the scattered light will transmit through the top surface. If a receiving device is placed to collect the scattered light, the defects will be imaged. Both surface and subsurface defects in the sample are detected simultaneously. So a question will arise: how to differentiate subsurface defects from surface ones? A software, differential interference contrast released by the National Institute of Health (NIH), is now available to in part solve the problem [28]. In addition, because defects are usually located at different depth, the images of scattering sites will be marginally out-of-focus which are slightly larger than the actual size of defects. Thus the TIRM to date experiences difficulty in accurately quantifying the depth/size of defects, which needs further investigation. Moreover, the TIRM requires that samples possess high quality surface so as to minimize the scattering of laser light on sample surface and the sample be transparent with respect to incident light, which restrains the TIRM from applying to broader fields. The TIRM is suitable only for polished or low surface roughness sample, especially those containing scratches/digs covered with the re-deposition layer and semi-finished optics in which the subsurface microcracks are not completely removed.

A modified TIRM, intensity-detecting TIRM (iTIRM), was recently developed by a research group in the Netherland as a tool for in-situ monitoring polishing process by measuring the surface roughness and subsurface damage of optical component being processed [150]-[154]. They have successfully incorporated the setup into the polishing to determine whether the polishing process can be stopped, which makes the polishing more cost-effective and time-saving and meanwhile guarantees the quality of manufactured optical parts. In addition, TIRM has been applied to the study of laser-induced damage to find out possible damage precursors [148].

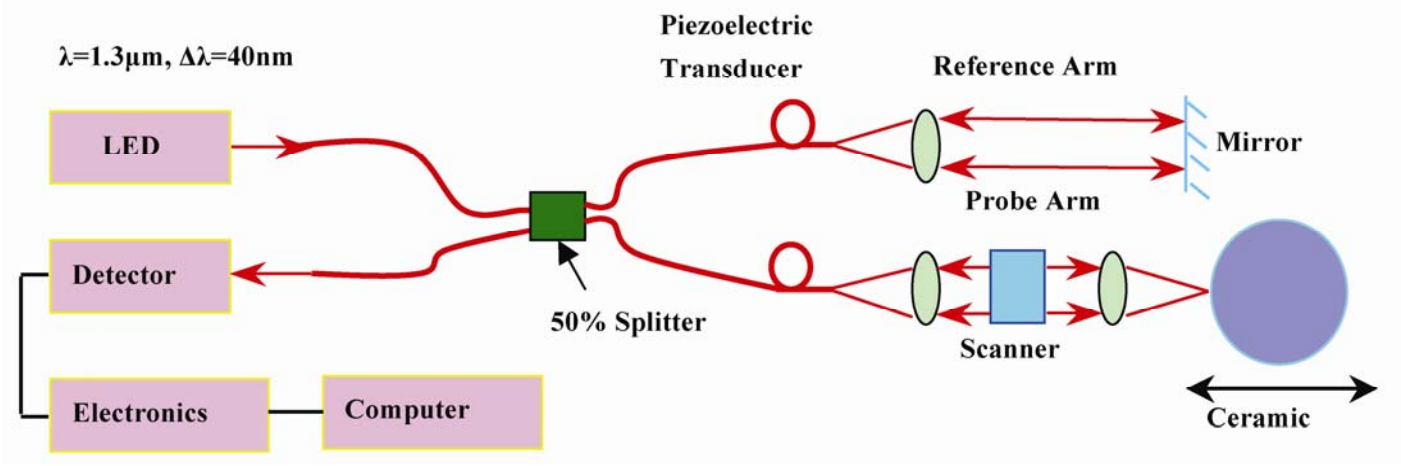


FIG. 8 Schematic representation of the OCT scanning system for detecting subsurface defects in ceramics. Longitudinal scanning is accomplished by translating ceramic samples with a stepper motor. The right half of the above picture constitutes a standard Michelson interferometer [164].

4.4 Optical coherent tomography

Optical coherent tomography (OCT) was first put forward for observing biological tissues, which can provide the cross-sectional photographs of subsurface structure of tissues; afterwards, the application of OCT was extended to engineering ceramic materials (Figure 8) [155]-[170]. Both coherent ultra-short laser pulses and low coherent light can be utilized as light sources in OCT to assess internal structure of samples. Unlike confocal microscopy whose resolution is limited by the numerical aperture, the OCT's depth resolution relies on the coherent length of light sources [157].

$$\left(\Delta L = 1n(2)\frac{2}{\pi}\frac{\lambda^2}{\Delta\lambda}\right)$$

Therefore, the low coherent light such as light emitting diode (LED) is preferable in order to improve the depth resolution. Because the light travels at different speed in samples from in the air, the refractive index of samples must be taken into consideration to obtain the actual physical depth in the samples. Using OCT, defects as deep as ~ 500 μm have been detected successfully and the typical vertical resolution of ~ 20 μm and ultra high resolution ~1 μm in air (equivalent to ~ 20/*n* μm in samples, n the material index of refraction) has been obtained [155], [165], [166]. Additionally, OCT, in principle, can be applied to the examination of stress-induced birefringence in the subsurface of materials for it is inherently sensitive to birefringence [170].

OCT shows promising future in imaging laser damage sites in optics as well. Incorporating the OCT into in-process and/or off-line monitoring apparatus to detect subsurface cracks resulting from high fluence laser one can gain the knowledge of damage initiation and growth, which is beneficial to damage mitigation [171]-[173]. The potential to examine defects in sample at long distance has been understood in biomedical field at first and extended to material processing research. Guss et al. [172] have constructed along working distance 3D OCT to remotely monitor the CO_2 laser post-processing of laser-damaged fused silica optics. In general, beneath craters resulted from high energy laser pulses are numerous cracks due to mechanical fracture. In order to repair these damaged sites, CO_2 post-processing is used because of laser heating effect. Whether or not the recovery is finished and completed needs off-line inspection. OCT having ability to image subsurface cracks is employed to in situ detect the presence of cracks after laser annealing. This setup works well at a distance of 50 mm and can image the cracks hundreds of microns in size.

4.5 Other techniques

Improved White Light Interferometer (WLI)

Steinert et al. [174]-[175] have reported that an improved white light interferometer, which is generally utilized to check the surface quality of a substrate, can be used to detect the lateral subsurface cracks. They have believed that most of subsurface lateral cracks are open and roughly parallel to sample surface and the incident light can be reflected slightly. Thereby, the depth of cracks is calculated through dividing the optical path difference (OPD) between surface and cracks by the refractive index of the substrate. This method can also detect the OPD due to the residual stress induced by manufacturing. Nevertheless, this method may be inappropriate when used to measure median cracks that are basically normal to sample surface and optically closed. As mentioned previously, median cracks dominate the strength of samples and extend far deeper below the sample surface while relatively shallow lateral cracks determine material removal and surface roughness.

Micro-particle coating

Sensor 21 Inc. [176] recently put forward a new inspection technique for SSD. A kind of coating containing luminescent micro-particles is applied to ground surface. The particles range from 1 nm to 5 μm in size. Larger particles stick to rougher surface and give out more intense light, which

matches the surface roughness of substrate. Thus, the surface cracks will be quickly and qualitatively pictured in terms of light intensity with charge-coupled device (CCD). This characterization technique is somewhat similar to dye impregnation [92], [93].

Quantum dots

In order to visualize subsurface in lapped optical parts, Williams et al. [177-179] have added quantum dots, a type of fluorescent particle ~ 8 nanometers in size, to slurry during lapping, which is in principle same as the methods of Baspiking [7], [180] and manufacturing fluid containing fluorescent dye penetrant [181]. Then the parts were observed with a wide field fluorescence microscope. If the part contains subsurface damage, the quantum dots penetrate and firmly embed in the surface of microcracks and can be imaged with the microscope. Once subsurface damage is eliminated, the quantum dots will not adhere to the surface of optical parts. The penetration depths of dots indicate the depth of subsurface damage, and the different depth of dots due to subsurface damage can be determined with confocal microscopy. However, the measured depth of quantum dots seems very likely to be smaller than SSD depth actually is since the dots probably are blocked not to arrive the tip/bottom of microcracks owing to wedged shape. This method can identify SSD as deep as 10 μm and have resolution of less than 0.5 μm significantly smaller than most nondestructive methods.

Micro-indentation technique

The surface of ground optics will be different from the bulk material in mechanical and physical properties. The microindentation method takes advantage of the different hardness of the surface layer to detect the depth of subsurface damage [182], [183]. Polvani and Evans [182] have used the microindentation to study the SSD of ground fused silica. They have found that the hardness of gradually increases with increasing the indentation depth to the bulk value of substrate. The thickness of the layer with degraded hardness can be referred to as the depth of the SSD. The phenomenon was also observed by Paehler et al. for ground silicon wafers [184] who determined and profiled the Yang's modulus by laser acoustics method in combination with step chemical etching. Moreover, Yang [185] systematically studied the influence of SSD on such parameters as maximum indentation depth, elastic modulus, etc. and found that the maximum indentation depth at greater SSD is deeper than that at less SSD under a given load. According to the mechanical theory that if the load imposed on the indenter is fixed, the larger the indentation depth is, the less hard the tested sample is, the Yang's results are, in effect, in accord with those of Polvani and Paehler. This fact is indicative of usefulness of hardness or elastic modulus as a tool of measuring SSD in ground optics. This technique can assess the SSD as small as several microns, but nonetheless the test results may not be reliable when applied to SSD less than 1 μm.

5 SUMMARIES AND OUTLOOK

The SSD is in itself statistically meaningful and more often than not the measurement of SSD depth relies strongly on the specific evaluating techniques. Accordingly it is not surprised that different methods yield different SSD depths [186]. Generally speaking, destructive method can give precise values of SSD depth because of high resolution relative to nondestructive techniques. Nonetheless, non-destructive evaluation methods for surface and subsurface damage are more expedite than destructive method, whereas most of the nondestructive techniques are of qualitative evaluation. In contrast, the destructive evaluation can provide direct, reliable, and quantitative information of the subsurface damage, thought it will render the sample unusable any more in some cases. We believe that some new techniques nondestructively and quantitatively assessing the sub-/surface damage will appear in future with the great progress in optical science, manufacturing and testing technology and other realms of science and technology. The goal of subsurface damage evaluation is to make manufacturing processes, specifically speaking, grinding and polishing processes more efficient and economical and to pave the way for achieving the ''perfect'' finished surface of optical components. The nondestructive techniques are relatively easily integrated into manufacturing streamlines as compared to the destructive methods, which maybe facilitate the in-situ testing of surface and subsurface damage during the manufacturing of optical components. As has demonstrated by Fahnle et al. [150]-[154], the TIRM has been applied to in-line monitoring the sub-/surface information in order to determine the endpoint of polishing process.

On the other hand, the testing of surface and subsurface quality will spur the emergence of novel manufacturing technologies such as deterministic microgriding, RAP processing, MRF polishing, elastic emission machining, etc. [12]-[20], [187], [188]. Some innovative grinding techniques are desired to minimize subsurface damage efficiently and new polishing processes to eliminate the subsurface damage while maintaining the ultra-precise surface form accuracy in order to produce the ''perfect'' surface as if created by God. Because the mechanical techniques cause the damage or stress in brittle materials, the defect-free manufacturing methods should be characterized by, to greater extent, chemical effects. Since chemical reactions occur on molecular or atomic level chemical manufacturing introduces no additional damage. Moreover, the deterministic manufacturing technologies are necessary to shorten the iterative procedure so as to make manufacturing more timesaving and cost-effective.

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