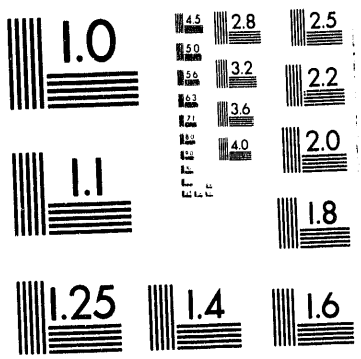
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A Historical Perspective on Fifteen Years of Laser Damage Thresholds at LLNL

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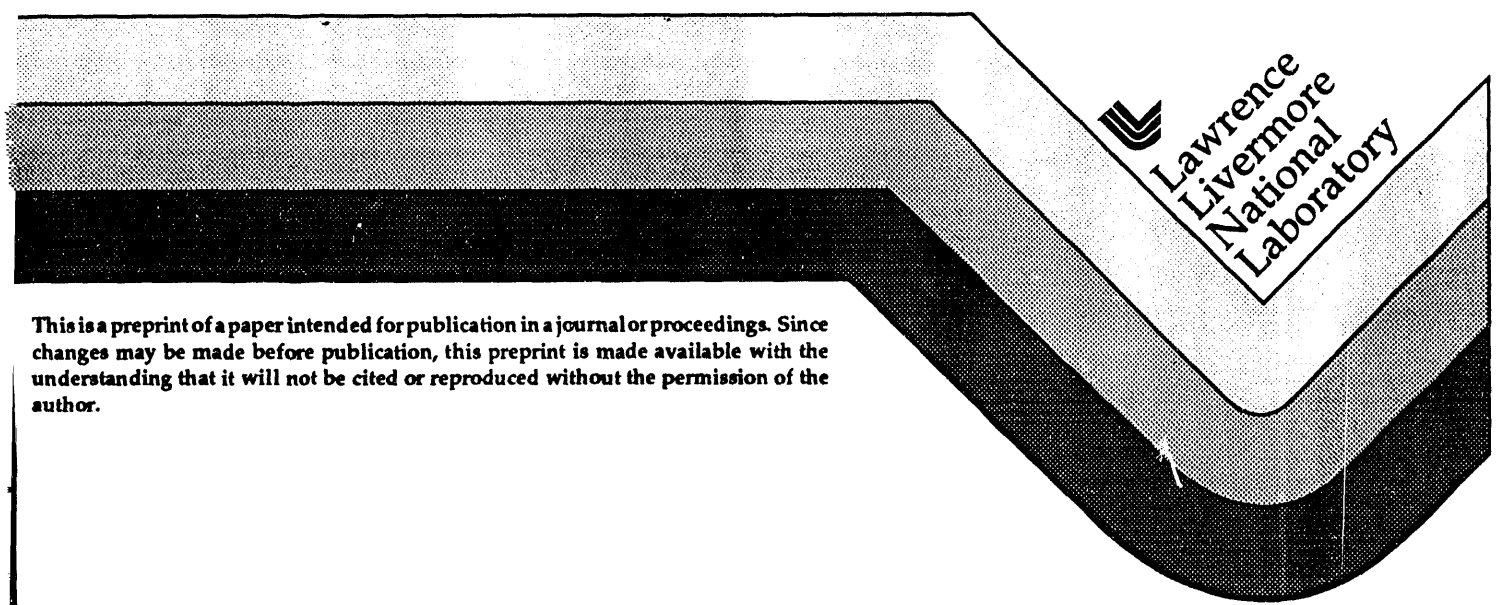

A historical perspective on fifteen years of laser damage thresholds at LLNL

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ABSTRACT

We have completed a fifteen-year, referenced and documented compilation of more than 15,000 measurements of laser-induced damage thresholds (LIDT) conducted at the Lawrence Livermore National Laboratory (LLNL). These measurements cover the spectrum from 248 to 1064 nm with pulse durations ranging from < 1 ns to 65 ns and at pulse-repetition frequencies (PRF) from single shots to 6.3 kHz.

We emphasize the changes in LIDTs during the past two years since we last summarized our database. We relate these results to earlier data concentrating on improvements in processing methods, materials, and conditioning techniques. In particular, we highlight the current status of anti-reflective (AR) coatings, high reflectors (HR), polarizers, and frequency-conversion crystals used primarily at 355 nm and 1064 nm.

1. INTRODUCTION

On a biennial basis we have in recent years published in these proceedings database summaries of LIDTs measured at LLNL.[1, 2, 3] These summaries, as well as many ancillary publications, have documented the development of high-threshold optical components for use in large-scale laser systems employed in the research of inertial confinement fusion (ICF). These progressively more powerful laser systems have included: Long Path (1970), Janus (1974), Cyclops (1975), Argus (1976), Shiva (1977), Novette (1982), Nova (1984), Beamlet (1994), and the proposed National Ignition Facility (NIF). The development of high-threshold optical components for these systems involved extensive collaborative efforts between LLNL and many commercial vendors, universities and laboratories both foreign and domestic. A partial list of these is shown in Table. 1.

We are engaged in an effort to ultimately publish a non-proprietary version of this database which will cover the spectrum from 248 nm to 1064 nm with pulse durations ranging from < 1 ns to 84 ns. We shall highlight here some of the more crucial elements in ICF laser systems shown in bold in Table 2. We will provide a perspective on the development of damage thresholds for these components during the past 15 years with particular emphasis on current thresholds at 355 nm (3ω) and 1064 nm (1ω) measured during the past two years. In order to provide a common footing for thresholds measured at a variety of pulse durations, we will present these results in terms of 3-ns values, scaled by appropriate, empirically-determined scaling factors. More extensive details of materials used in each of the categories may be found in reference 3. Generic descriptions of our damage test facilities and capabilities can be found in reference 4.

We have conducted damage tests with PRFs ranging from single shots up to 6.3 kHz. Most of our recent tests have been conducted at a PRF of 10 Hz. Evidence of threshold enhancement has been demonstrated at these proceedings over the last few years utilizing conditioning irradiation in which the sample is irradiated by gradually increasing fluences. Fig. 1 displays the different methods of test irradiations which are specified in our database for

Acton	Deposition Sciences	Inrad	NSG	SPAWR
AFWL	Desag	Intermedics	NWC	Spindler & Hoyer
Airtron	Design Optics	Itek	OCLI	Stanford (U)
Alfred (U)	Diamonex	Japan	OI	TecOptics
Allied	DuPont	Kigre	Optico Glass	Thin Film Coat
American Thermal	Dynasil	Kirtland	Optics Plus	Tinsley
Applied Optics	Echantillon	Kodak	OptiFab	Toshiba
Arizona (U)	Edmund	Labsphere	Optochemical	Trans World Opt.
AT&T	Epner Technology	Lambda Airtron	Optovac	Ukraine (ISC)
Aurel	Epoxy Technology	Lambrecht	Oriel	Ultramet
AWRE	ERIM	Lasermetrics	Osaka (U)	Unertl
Balzers	Feroxcube	Laser Optics	Pacific Optical	Union Carbide
Battelle Columbus	Fish-Schurman	Laser Power Optics	Pacific West S.	United Lens
Battelle PNL	France	Lensbond	Perkin Elmer	US Paint
Bond	Fujian	Lightning Optical	Phillips	USSR
Broomer	GE	Limeil	PMS	UTOS
Burleigh	GEC	Litton	Polycast	Virgo
Canon	Gentec	LLNL	Polymer Corp	WDQ
Cascade Optical	Germany	Loctite	Quartz & Silice	Westinghouse
CCI	Gesänger	Matra	Rochester (U)	Willis
Central Florida (U)	Glendale	Meller	Rocky Flats	Wisotzki
China	GM Assoc	MMM	Russia	ZC&R
Coherent	Halocarbon	Moscow (U)	Russia (IAP)	Zygo
Continental	Harshaw	Mykroy	San Francisco (U)	
Corning	Heraeus	NBS	Schott	
Crystal Technology	Honeywell	New Mexico (U)	Scientific Coating	
CVD	Hoya	Newport	Shandonz	
CVI	Hughes	Norland	Shanghai	
Davis (U)	Hyperfine	Northrop	Showa	
Deacon	IBM	NRL	Spectra-Physics	

Table 1. Partial list of commercial vendors, universities, laboratories, and countries that have contributed to the development of high-threshold optical materials for LLNL.

Coatings	**ANTI-RELECTIVE (AR), HIGHLY REFLECTIVE (HR), POLARIZERS,** single and multiple layers
Substrates	surface and bulk thresholds for the above elements, lenses, optical rotators and waveplates, laser glasses, filters
Crystals	surface and bulk thresholds for **FREQUENCY CONVERSION,** laser hosts
Gratings	reflective, transmissive
Metals	bare metals, overcoated metals
Miscellaneous	plastics, ceramics, epoxies, diffusers, liquids, paints, teeth

Table 2. Types of components tested for laser induced damage thresholds at LLNL.

frequency conversion crystals. The methods on the right indicate situations under which thresholds may be improved (including pre-irradiation thermal annealing). Our current primary irradiation methods for conducting damage tests are at 10 Hz with 600 shots of the same fluence (S/1), or 600 shots of ramped-up fluence (R/1).

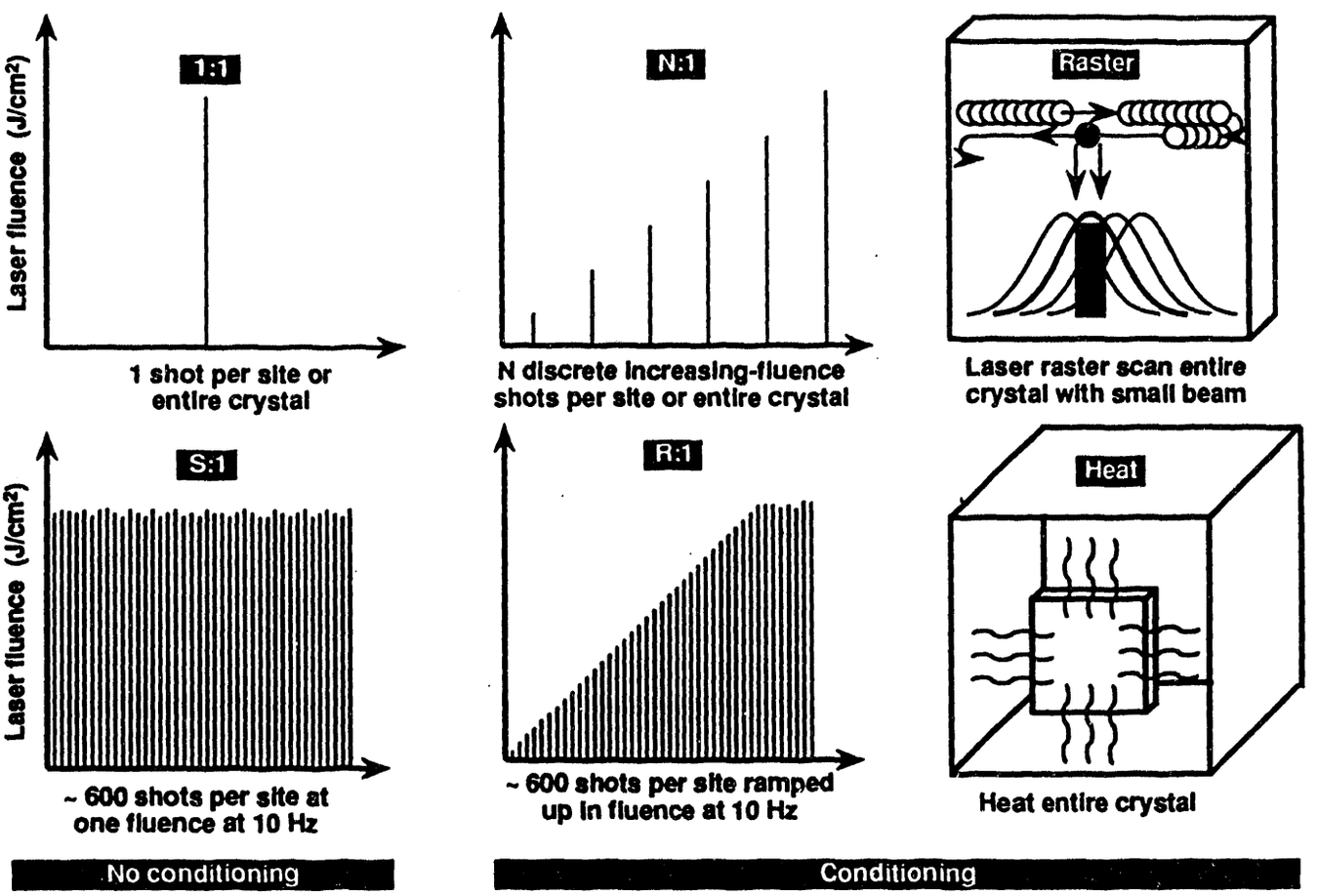

Fig. 1. Definitions of the different types of irradiation and conditioning methods (in this case for KDP crystals).

2. OPTICAL COMPONENTS

2.1 Anti-reflective (AR) coatings

Fig. 2 summarizes the results of *all* damage tests that we have conducted at 1ω on AR coatings. In this, as well as in subsequent figures, small circles will represent unconditioned irradiations (usually 1/1 in the early years, S/1 in the later years); large circles imply conditioning by N/1, R/1 or rastered irradiation. However, the figure does not show a one-on-one comparison of conditioned vs. unconditioned thresholds for specific samples. We conducted most of our recent tests with conditioned irradiation which typically provided a small amount of threshold improvement. This was probably due to cleaning and expulsion of volatile solvents. However, we seldom observed improvement by factors of two or three that has been common with HRs and polarizers (see below). Fig. 3 shows a corresponding, but smaller, database of such tests conducted at 3ω. The 3ω work lagged a few years behind that at 1ω and for a while waned as our testing capabilities changed. The legends in each case specify the ranges of pulse durations used in the

tests. In order to facilitate comparison of the development of damage thresholds over wide ranges of pulse durations for thousands of tests, we have scaled all the thresholds to 3-ns values by a scaling factor of $\tau^{0.35}$. This is an empirically derived factor and varies some between AR, HR, and polarizer coatings. Our efforts followed two separate paths.

In the early years (1978 to 1983) of our research in high-threshold optical coatings, we exerted our greatest efforts in trying to develop acceptable AR coatings for use at 1ω. At that time these proved to be the most damage-prone elements in high-fluence laser chains. The conventional method employed multi-layer, alternate coatings deposited by e-beam evaporation. These typically consisted of a combination of a high-index-of-refraction material and silica. During those years we have presented at these proceedings the results of improvements in laser damage thresholds employing one or more of the following techniques: choice of substrates[5], choice of high-index material[5,6], half-wave-thick undercoats of silica (or another low-index material) between the AR stack and the substrate (barrier layer)[6,7], deposition parameters (temperature, O_2 pressure, deposition rate), and modified layer designs (layer thickness, layer numbers)[6]. The average annual improvement in thresholds by these techniques ranged on the order of only 10 to 15%. Histograms of the choice material combinations for such e-beam deposited coatings are shown in reference 3 for wavelengths ranging from 1064 nm to 248 nm.

LLNL, in conjunction with commercial and government laboratories, also experimented with a variety of more exotic and often vendor-proprietary techniques to generate AR properties on assorted substrate types. We conducted damage tests on gradient-index films[8], leached films, phase-separated glass, neutron-tracked glass, metalo-organic-deposited (MOD) films, liquid films, Schroeder-process films, neutral-solution coatings[9], and solgel coatings[10]. Although it has not been practical to identify these types of AR coatings in Figs. 2 and 3, in general we found that many of the high-threshold samples stemmed from this latter set of processes. However, in most cases these technologies were not able to be extended to production optics. This was for a variety of reasons: the optical or damage properties were not uniform, the AR surfaces had high densities of defects, the processes could not be applied to large optics, the AR surfaces were very fragile, or the processes were very expensive.

Between 1984 and 1991 we concentrated almost exclusively on the solgel process, applied either by dip or spin coating. Our earlier endeavors did not necessarily yield thresholds as high as those of the better e-beam coatings, but careful attention to clean processing ultimately resulted in AR coatings having among the highest thresholds, rather than the lowest, for coated optical elements.[11,12] The fragile nature of solgel proved not to be a significant problem. Coatings could in fact be spray cleaned *in situ*.[13] If contaminated or damaged, the entire coating, often on the order of 1 m in size, could simply be wiped off and recoated. This would be at a cost of a few hundred dollars as opposed to tens of thousands of dollars required to polish off and recoat an e-beam-deposited AR. By the late 1980s we resumed 3ω coating fabrication and testing. These also ultimately had thresholds higher than other 3ω coated optics.

Most of our AR efforts in 1992 and 1993 concentrated on an assortment of experimental techniques which included ARs fabricated with a meniscus coater, including the use of sec-butanol as a suspension medium[14], ammonia-treated solgels, dip- and thermally-evaporated Teflon[15,16] and silicone coatings, the use of organic polymer bindings[17], and the treatment of coating contamination in a hostile target-chamber environment. These tests were conducted at both 1ω and 3ω yielding a large spread in thresholds. The very high thresholds observed in 1990 were not repeated. That was because those coatings represented the state-of-the-art, production coatings applied routinely on the optics used in the Nova laser facility. Most tests in 1992 and 1993 involved assorted experimental techniques which usually had somewhat lower thresholds.

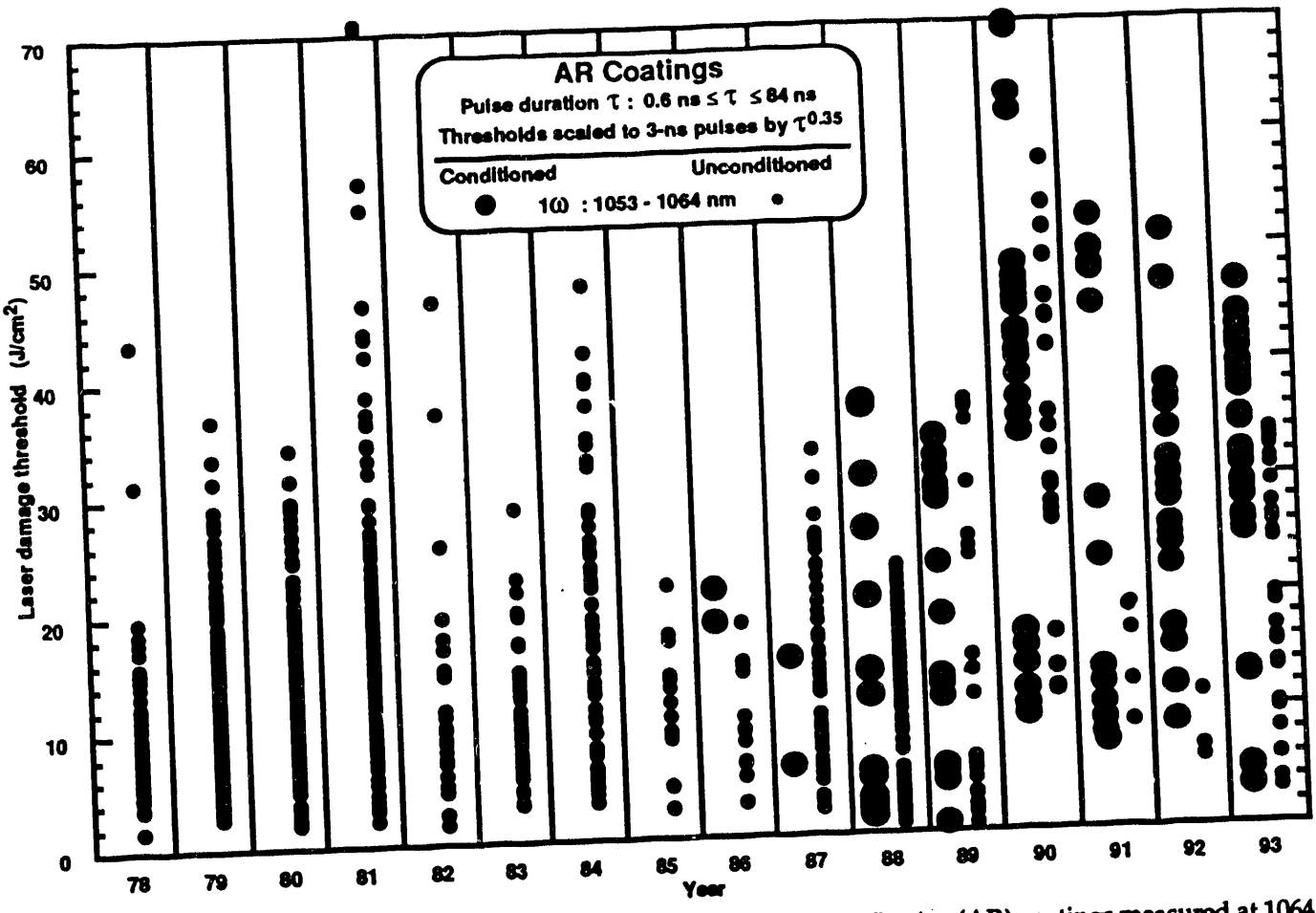

Fig. 2. Conditioned and unconditioned damage thresholds of all anti-reflective (AR) coatings measured at 1064 nm (1ω), scaled to 3-ns values by $\tau^{0.35}$.

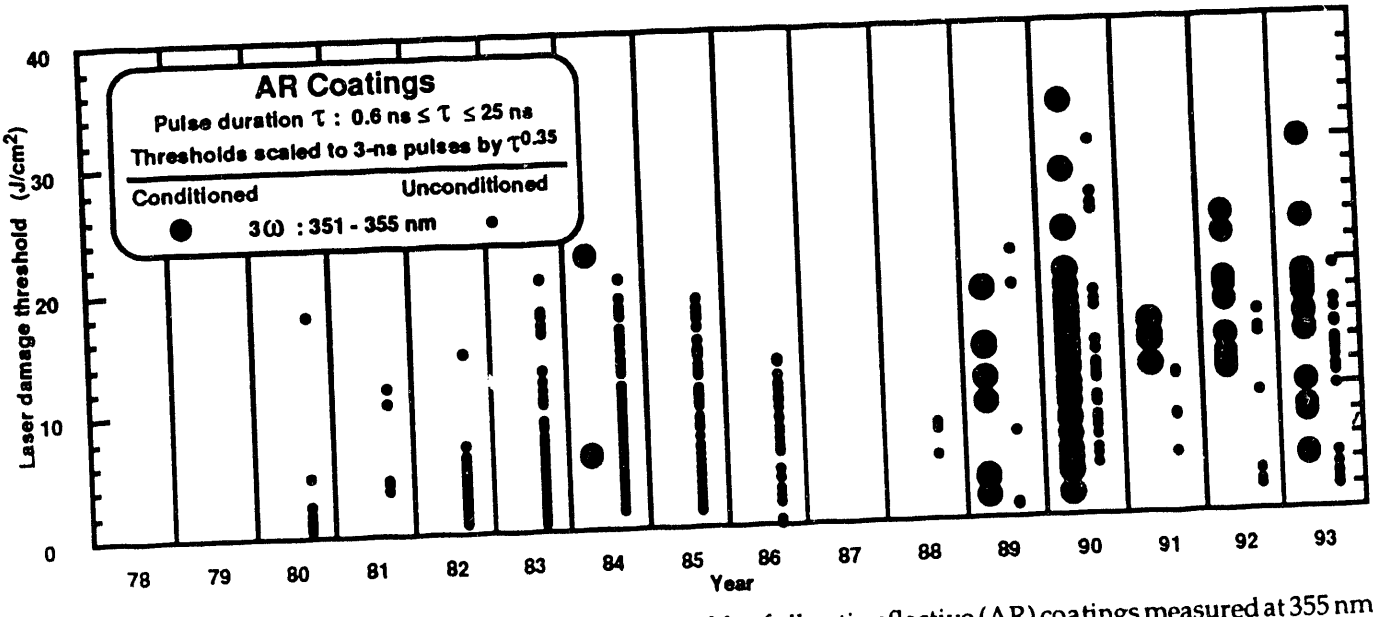

Fig. 3. Conditioned and unconditioned damage thresholds of all anti-reflective (AR) coatings measured at 355 nm (3ω), scaled to 3-ns values by $\tau^{0.35}$.

2.2 Highly reflective (HR) coatings

Figs. 4 and 5 show respectively all of our damage testing conducted on HR coatings at 1ω and 3ω. The relationships of materials to damage thresholds at these and the other harmonics are again provided in detail in reference 3. The same comments regarding conditioning and scaling of the thresholds to 3-ns values apply to HRs as were noted for ARs.

All of the developmental work in high-threshold HR coatings between 1978 and 1986 was conducted with unconditioned irradiation (almost exclusively 1/1). In these years we worked primarily in collaboration with commercial vendors to fabricate multi-layer, e-beam-deposited coatings. To a lesser degree, and also with less success, we investigated sputtered and ion-assisted coatings. We conducted many of the same types of parameter variations mentioned for e-beam-deposited AR coatings. These included the choice of high-index material, multiple high-index materials, half-wave-thick overcoats of silica (or another low-index material) on top of the HR stack, deposition parameters (temperature, O_2 pressure, deposition rate), and modified layer designs (layer thickness, layer numbers).[5,18,19] Coating designs were also modified to provide desired multi-chroic optical characteristics at 1ω, 2ω and 3ω. The fact that the earlier tests were conducted only with single pulses led to some erroneously high thresholds in coatings, particularly those made with titania.[20] Single-shot damage in such HRs was often so subtle that it was imperceptible with 100x Nomarski microscopy. However, multiple shots subsequently led to the rapid growth of that subtle damage to readily visible proportions. This prompted us to concentrate more on materials such as tantala and hafnia at 1ω and scandia and various fluorides at 3ω.[18]

With construction of the Reptile damage facility in 1986, we conducted virtually all of our damage tests from 1986 to 1991 using rep-rated pulses ranging in PRFs from 10 to 120 Hz. These proved to be effective "lifetime" tests of the optics since a one-minute irradiation on Reptile could actually represent a year or more of shots conducted with a Shiva or Nova laser. It was in this time span that we began to pursue conditioning of HRs as an offshoot of earlier KDP work. It turned out, that even in the late 1970s, we had observed that the bulk damage thresholds of KDP crystals could be raised by gradually increasing the fluences of the test laser pulses from very low levels (see below).[21,22] With a single-shot laser this was a tedious, time-consuming operation. Using a rep-rated test laser we were able to conduct such irradiations with hundreds of shots of increasing fluences in just one minute rather than tens of shots in hours. The ramping was accomplished by varying the laser fluence with a remotely-controlled rotation of a waveplate. We designated this as R/1 irradiation as compared to the earlier N/1 irradiation of numerous single shots.

The improvements in damage thresholds by the conditioning of 1ω HR coatings, particularly those made of hafnia/silica, was so promising that a great deal of effort was spent during the subsequent years. Our research showed that (1) the rise in thresholds typically ranged by factors of two to three, (2) was permanent over a time span of months, (3) could be accomplished to some degree by a few full-aperture shots of the Nova laser or by rastering with a small rep-rated laser.[23,24] We established that the primary source for the onset of damage in the HRs stemmed from nodular defects deposited during the coating process. This prompted us to begin a study of such defects using Atomic Force Microscopy (AFM).[25] Concurrent with the conditioning efforts of HRs fabricated by conventional e-beam deposition for immediate deployment on Nova, we also began to investigate some experimental techniques. Initial results of damage testing of multi-layer solgel HRs proved to yield only marginally acceptable thresholds.[26] On the other hand, we experienced some of our highest thresholds by first depositing and repolishing thick dielectric layers on a substrate before the final HR stack was deposited. This was accomplished successfully on dielectric substrates, metal substrates and metal layers in support of LLNL's free-electron-laser program.[27]

During 1992 and 1993 we continued to emphasize the AFM study of nodular coating defects and the

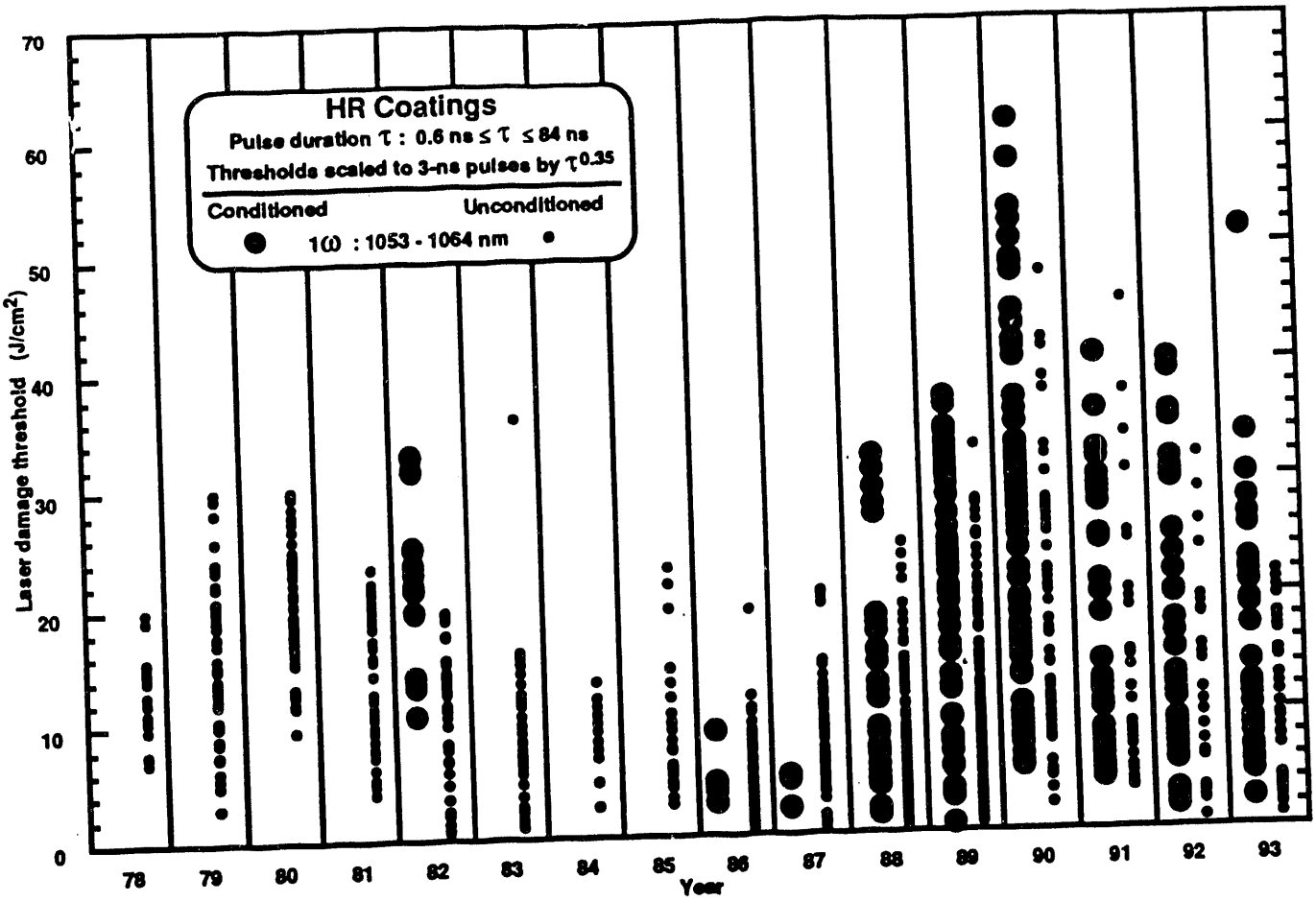

Fig. 4. Conditioned and unconditioned damage thresholds of all highly reflective (HR) coatings measured at 1064 nm (1ω), scaled to 3-ns values by $\tau^{0.35}$.

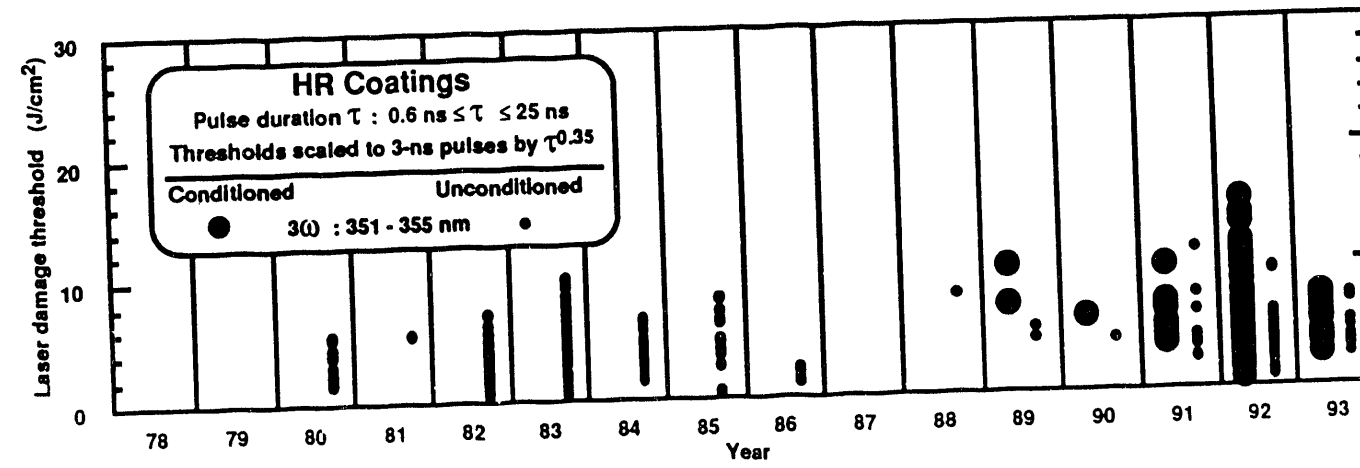

Fig. 5. Conditioned and unconditioned damage thresholds of all highly reflective (HR) coatings measured at 355 nm (3ω), scaled to 3-ns values by $\tau^{0.35}$.

improvements of their thresholds with conditioning.[28,29,30] The threshold requirements of future laser systems necessitate that current defect-laden HR coatings need to be conditioned to comfortably survive high laser fluences. The preferable alternative would of course be to eliminate the defects altogether during the coating process. This has been under study at LLNL by a variety of techniques, including baffling and electrostatic repulsion, to reduce the effects of spatter.[31] The spread in damage thresholds shown in Fig. 4 for the last few years has actually not changed much. The highest thresholds achieved in 1990 with the polished, undercoated, thick layers has not been duplicated by conventional e-beam coatings. However, it should also be noted that most of the low threshold HRs shown for recent years are not representative of routinely achievable good optics. These samples included further experiments with sputtered HRs, ion-assisted coatings, and vendor qualification tests. Experimental solgel HR coatings fabricated by spin coating or meniscus coating have not yet yielded cosmetically clean production-grade samples.[14] However, they have shown encouraging signs of improving in cleanliness and thresholds, particularly with the use of PVA binders.[32]

We expended the greatest amount of effort in almost a decade in addressing damage issues to 3ω HRs. Depending on the applicable scaling factors we have observed 3-ns thresholds approaching 15 J/cm^2. However, cosmetically clean coatings usually fell at half that level with little or no improvement attributable to conditioning. Moreover, because of the sensitive design and fabrication criteria for high-angle-of-incident HRs (≥45°), such mirrors typically have had even lower thresholds. The preferred high-index materials are still scandia and assorted fluorides as was the case in the early 1980s for both 3ω and 4ω coating development.[18,33]

2.3 Polarizers

Polarizers have become one of the more critical coating components in large-aperture laser systems. Fig. 6 shows all of the tests that we have conducted on polarizers at 1ω, scaled to 3-ns values by $\tau^{0.35}$. We have conducted virtually no polarizer tests at the other harmonics since they are not employed in our large lasers. Polarizers present an added complication to those of HRs at high angles since they cannot simply be designed to sit somewhere near the middle of a broad reflection peak. They must, in fact lie near an edge so as to transmit most of the P-polarization while reflecting most of the S-polarization.

Between 1978 and 1988 we conducted a relatively small number of polarizer damage tests. Commercial vendors had little interest in devoting any significant resources in a relatively complex problem with less financial payback than HRs. Moreover, in those earlier years, polarizers were typically situated in low-fluence positions in the laser chain so that they did not represent a significant problem as far as damage was concerned.

However, beginning in 1989 to the present, we have engaged in a concerted effort to improve both the optical performance and damage resistance of e-beam-deposited, multi-layer polarizers. This work piggy-backed on to our HR coating efforts so that we concentrated primarily on hafnia/silica coatings with just a few tests on tantala/silica samples. Except for some low-threshold coatings, evaluated for potential coating suppliers, our polarizer research involved many samples from only a few commercial vendors. The thresholds therefore showed more consistency since only small perturbations were usually attempted in optimizing coating runs. This was particularly evident by the relative spreads of conditioned and unconditioned thresholds. As a whole, conditioning of nodular defects improved damage thresholds by a factor greater than 2.[23] However, because of the sensitivity of coating designs at Brewster's-angle incidence, polarizers have not achieved thresholds as great as those of normal-incident HRs. This sensitivity was particularly noticeable for tests conducted in 1990. These samples, both experimental ones and production-run witnesses, showed a pronounced drop in thresholds from the previous year. Careful attention to the minimization of nodular defects during the coating runs has brought thresholds back up. However, we have not been

able to completely achieve the high-threshold consistency of the 1989 coatings. Subsequent retests of these original samples verified that they did indeed have high thresholds and were not just subject to a possible change in calibration of the damage testing experiment.

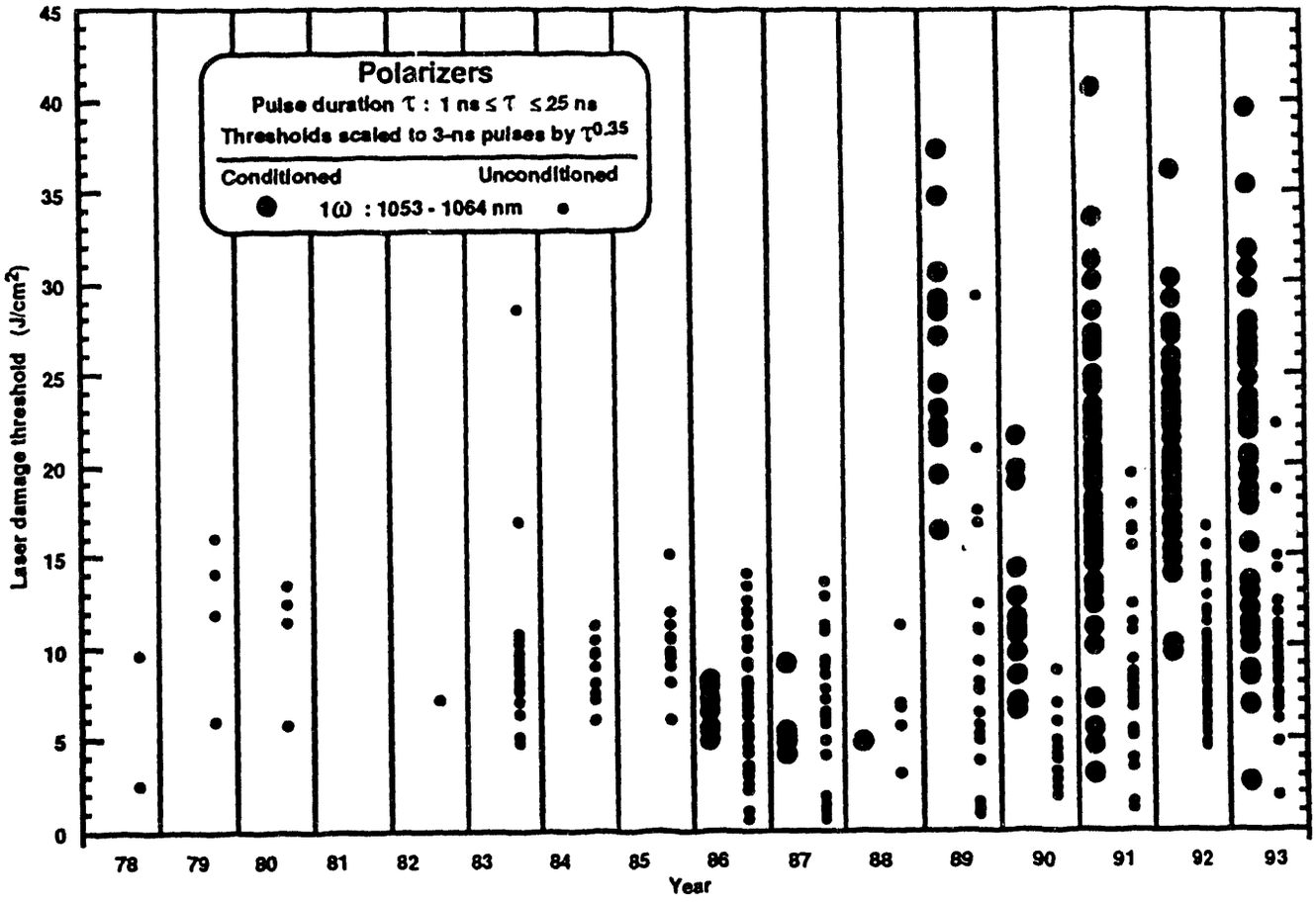

Fig. 6. Conditioned and unconditioned damage thresholds of all polarizers measured at 1064 nm (1ω), scaled to 3-ns values by $\tau^{0.35}$.

2.4 KDP and KD*P crystals

Over the span of 15 years we have conducted more damage tests on KDP and KD*P crystals than any other unique optic. Moreover, the actual data volume was made larger because (1) we have typically identified damage by its location at the front surface, rear surface, and within the bulk material, and (2) we routinely conducted both conditioned and unconditioned damage tests. Improvements in the diamond turning of the crystal surfaces and solgel AR coatings have resulted in relatively high surface-damage thresholds. Hence, the failure mechanism of recent samples usually stemmed from damage within the bulk material. Fig. 7 shows all of the conditioned and unconditioned threshold measurements that we have conducted on bulk material at 1ω and 3ω since 1986. We have again scaled the thresholds to 3-ns values, but by a scaling factor of $\tau^{0.5}$. The figure does not distinguish between KDP and KD*P.

Prior to 1988 the damage thresholds of KDP crystals varied considerably from sample to sample. These crystals typically had moderate to high densities of pre-existing bulk defects. The number of such visible defects usually

increased with laser irradiation or grew larger with fractures on the order of several hundred μm. We do not show any of the bulk threshold measurements prior to 1986 in Fig. 7. However, even in these early years, as noted above, we observed significant improvements in thresholds both by laser conditioning and thermal conditioning.[21,22,34]

From 1988 to the present we have observed steadily rising thresholds. This was attributable primarily to improved crystal-growing techniques employing ultrafiltration to produce crystals with bulk materials virtually free of any visible defects ≥ 5 μm in size.[35] Fig. 7 shows that at 1ω conditioning of KDP improved the threshold by a factor of 2.1 over unconditioned values.[36] We observed even greater improvement by thermally annealing KDP crystals at temperatures ranging from 135°C to 175°C.[37] On the other hand, some of the newest KDP crystals had such inherently high thresholds that they required no conditioning at all. At 3ω we did not observe improvements of the same degree as at 1ω, both with laser and thermal conditioning. Nevertheless, KDP thresholds would still exceed large-laser requirements.

KD*P showed comparable factors in improvement in 1ω thresholds with conditioning, but began at nominally 20% lower unconditioned thresholds. Thermal conditioning for KD*P was more restrictive since the crystal undergoes a phase transition at about 130°C for 80% deuterated KD*P. Thus, we observed only marginal improvements in 1ω thresholds with low-temperature thermal conditioning alone. At 3ω thermal conditioning to KD*P was essentially ineffective. For KD*P crystals that will be exposed to high-fluence, 3ω, laser irradiation, the crystals will have to be baked for longer times and/or be laser conditioned with a full-area beam on a large laser system or be rastered in stages with a small test laser.

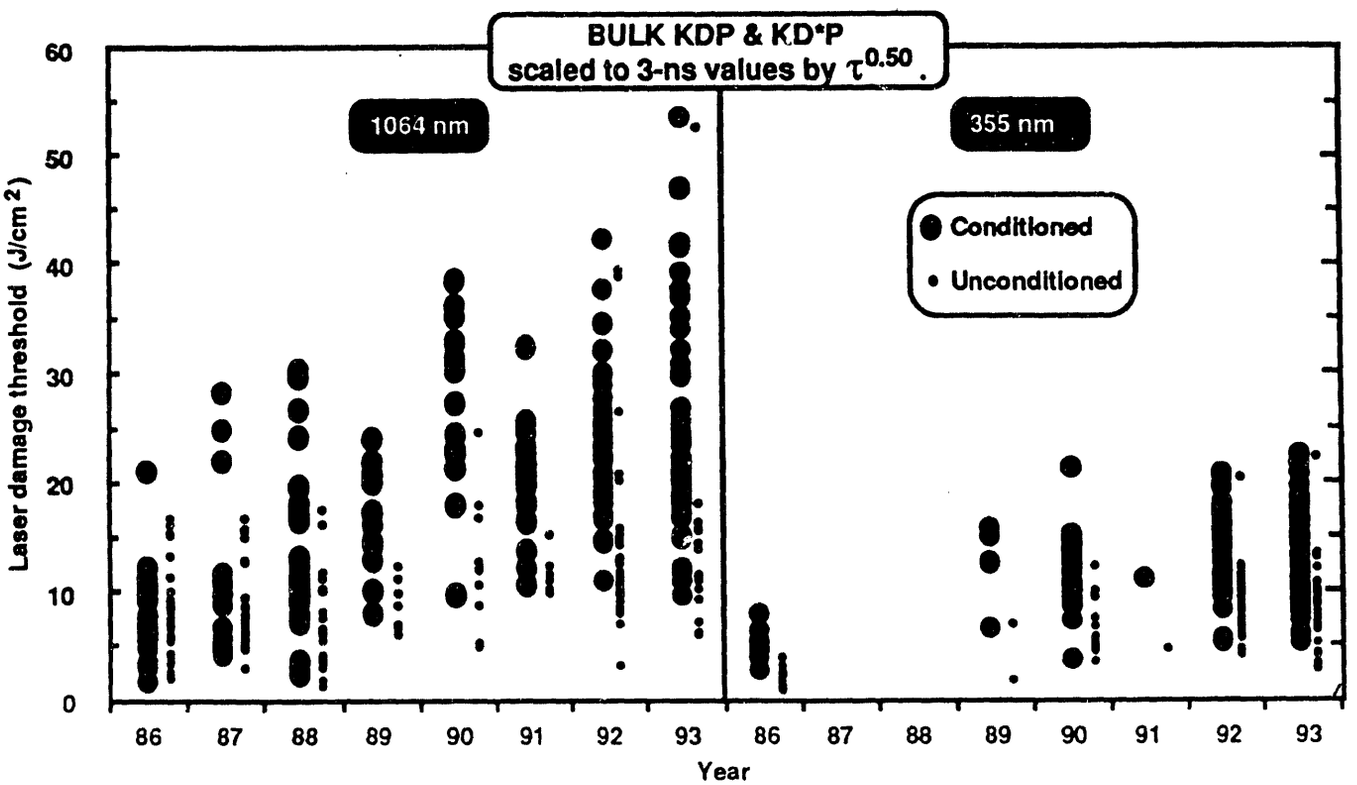

Fig. 7. Conditioned and unconditioned bulk damage thresholds of all KDP and KD*P crystals measured at 1064 nm (1ω) and 355 nm (3ω), scaled to 3-ns values by $\tau^{0.5}$.

3. STATUS OF CONTEMPORARY AND FUTURE OPTICS

LLNL is currently engaged in developing a conceptual design for a NIF laser. Not only must the components for this laser meet strict optical requirements, but very often the actual design revolves around the damage thresholds of several critical elements. In Fig. 8 we show an early proposal for the conceptual design fluences of various optics required on NIF. These have been normalized to a scaled pulse duration of 4.8 ns. The solid curves represent pulse-duration scalings of 1ω (shaded) and 3ω (black) thresholds for each respective optic type. Only the 3ω threshold of KD*P does not meet the strict interpretation of required damage threshold on NIF. However, as noted above, these are expected to improve with a combination of thermal and laser conditioning.

It should be noted that, not only are these thresholds based conservatively on the lowest measured value for an optic of recent vintage, but also often on a very strict interpretation of damage. Hence, a threshold has often been assigned based on individual damage pinpoints ≤ 10 μm in size. Since most optics typically already have pre-existing

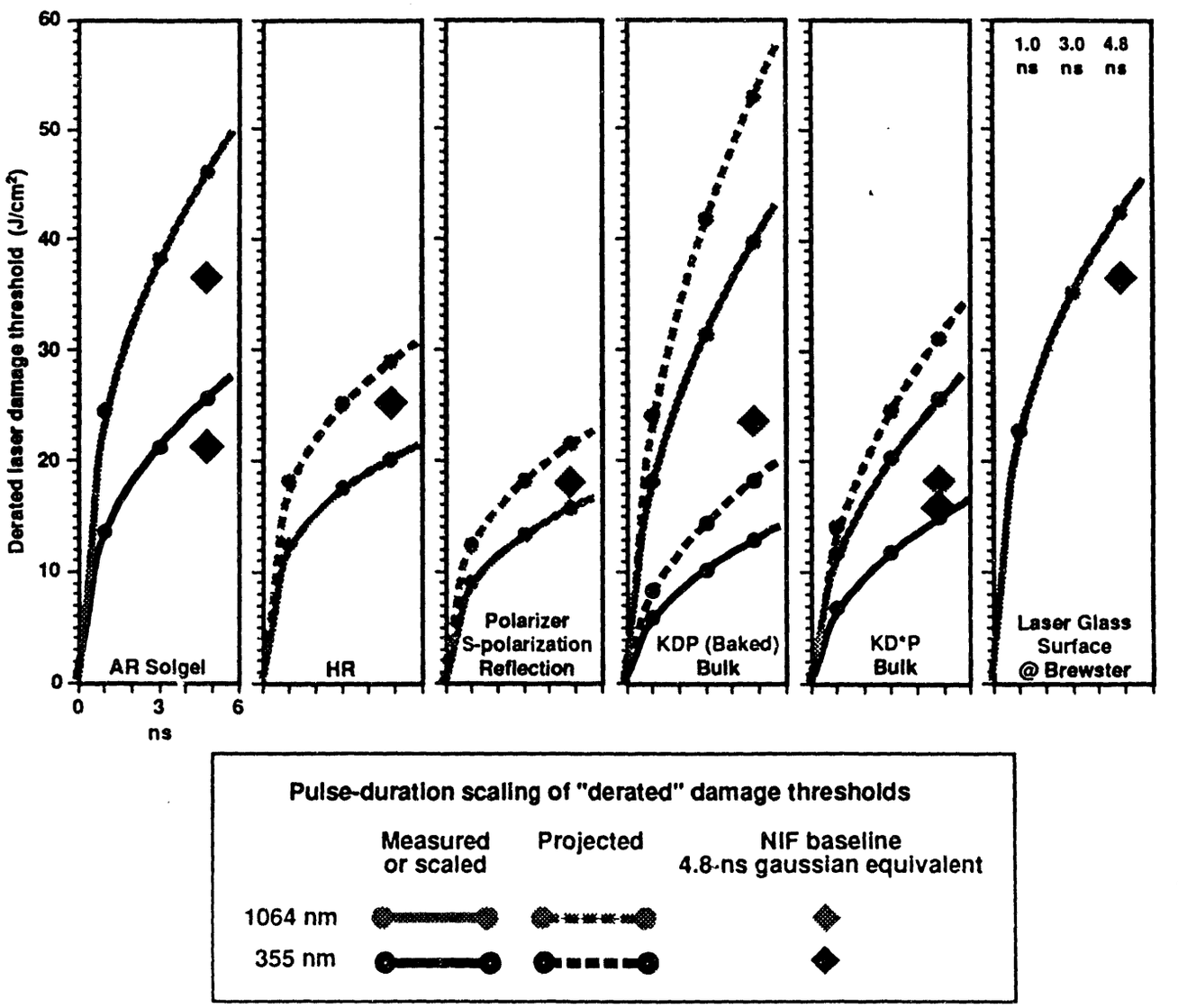

Fig. 8. Scaled damage thresholds and requirements at 1064 nm and 355 nm for crtical optical elements in a National Ignition Facility (NIF).

defects of such dimensions, a few more would not actually impact the performance of the optic. By our strict definition of damage, every optic on the current Nova laser is damaged. The dashed lines in Fig. 8 represent thresholds that have already been obtained for many samples and (1) can realistically be expected to be obtained in the future for all optics, or (2) would exhibit such subtle damage morphology that the damage would not affect the laser performance. However, in our testing, we will continue to adhere to our strict definition of damage since we only test a relatively small area of a sample. We need to be confident that the test area or volume is always conservatively representative, not only of the entire sample as a whole, but also of other samples fabricated under comparable conditions.

All of the evidence from the past two years has led us to conclude that two important caveats must be adhered to in order to achieve consistently high threshold optics in large laser systems. Defects, whether they resided on substrates, were deposited during coating, were grown within crystals, or were generated during subsequent handling and cleaning must be kept to an absolute minimum. Quality assurance must be in force throughout the fabrication, shipping, cleaning, installation and handling processes to guarantee that such high standards of cleanliness in laser optics are never compromised.

4. ACKNOWLEDGMENTS

During the past fifteen years a great number of people have made important contributions to the materials development and laser damage work of the ICF program at LLNL. The authors wish to acknowledge in particular the devoted efforts of their colleagues Dave Milam and Ted Hildum.

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