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14. ABSTRACT We optimize the properties and fabrication process of fiber Bragg gratings for sensors and for stabilization of semiconductor lasers. We investigate the effects of boron and titanium doping on thermal sensitivity. We find that gratings written through the polymer coating are stable to high-intensity infrared light traveling in the core of the fiber. These gratings are designed for wavelength locking of semiconductor lasers used for pumping erbium-doped fiber amplifiers. We find that hydrogen-loaded fibers have reduced mechanical strength compared to fiber gratings written without hydrogen. We also investigate the effect of thermal annealing on the strength of fiber gratings. We find that annealing at 230 degrees C slightly improves the fiber's mechanical strength, while annealing hydrogen-loaded fibers decreases the fiber's mechanical strength. Bending sensitivity of gratings is measured.					
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FINAL TECHNICAL REPORT

Final technical Report:0002AC for the period 01 Sept 1999 to 31 Oct 2000

Project Title: (STTR Phase II) "Novel Fabrication of Ultra-strong Thermally Stable Gratings through the Coating of Optical Fibers with UV Light"

Program Manager: Dr. Howard Schlossberg

Contract number: F49620-99-C-0059

Principal Investigator: Dr. Dmitry Starodubov

Institution Name: **Note: change of address!**

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Date Submitted: April 5, 2001

Personnel Supported: Dr. Dmitry Starodubov, Dr. Victor Grubsky

Change of Objectives: None

Patents filed: None

New Discoveries or Inventions: None

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Fabrication of Ultrastrong Fiber Bragg Gratings Written Through the Polymer Coating of Optical Fibers

The purpose of the STTR program is to help small companies get started and become commercially successful. This STTR grant has been enormously successful not only in that it has enabled D-STAR Technologies, Inc. to produce valuable scientific results, but it has enabled the company to establish itself, obtain external funding, and grow into a modest-sized company. This STTR grant to D-STAR Technologies, Inc. allowed it to grow from a company of 4 people in a rented laboratory into a thriving company of 40 people (at this writing) with its own 12,500 square foot production facility in Long Beach, California.

D-Star Technologies, Inc. is now shipping products in quantity to customers. Its gain-flattening filters are also being designed into the products of first-tier companies in Italy, England, and the United States. Its pump stabilizing gratings written through the fiber's polymer coating will make their commercial debut this summer.

A short history:

D-Star Technologies, Inc. was formed in 1998 by Prof. Jack Feinberg and Dr. Dmitry Starodubov with a quarter million dollars of "angel" funding from Ortel Corporation (now part of Agere). D-STAR received a Phase I STTR grant from the AFOSR in the Fall of 1998, and a Phase II grant for \$0.5M from the AFOSR in the Fall of 1999. In August of 2000 D-STAR completed its first round of funding for \$5M from Redpoint Ventures, a venture capital firm with offices in Los Angeles and Menlo Park. D-STAR leased a 12,500 square foot production facility in Long Beach, California and began production of fiber optic components in January, 2001. Sales for the first 3 months total \$0.5M. The expected revenue for the calendar year 2001 is \$10M.

D-STAR Technologies, Inc. makes components for fiber optic amplifiers. Specifically, D-STAR makes gain-flattening filters based on long-period gratings. Also, D-STAR Technologies, Inc. has patented a novel technology for writing Bragg gratings through the coatings of optical fibers. These gratings are ideal for pump stabilizers and especially for submarine applications, where mechanical strength and reliability are key issues. The physics of writing these pump stabilizing gratings in optical fibers was the focus of this STTR project.

Below we summarize the principal scientific achievements of this STTR project.

Principle scientific achievements:

- 1) Optimization of fabrication of fiber gratings written through polymer coatings.
- 2) Determined grating stability under light exposure.
- 3) Determined grating reliability under mechanical stress.
- 4) Determined design constraints for Bragg gratings for locking pump lasers.

1) Optimization of fabrication of fiber gratings written through polymer coatings in fibers with no hydrogen loading

We optimized the properties and fabrication process of fiber Bragg gratings for two applications: i) sensors and ii) stabilization of semiconductor lasers. Issues of fiber design, grating properties and grating reliability were addressed.

Gratings written in different fibers were compared for thermal sensitivity. We tested gratings written in high-NA fiber, a fiber having a Ti-doped cladding layer, and boron co-doped fiber. The high-NA fiber is manufactured by Fibercore, Ltd. (England), and is used by the US Navy in their towed sensor arrays. The fiber with 7% Ti in the cladding is designed to have a very low thermal expansion of the cladding. The fiber with a high boron concentration is used to make gratings for stabilizing 980-nm semiconductor lasers.

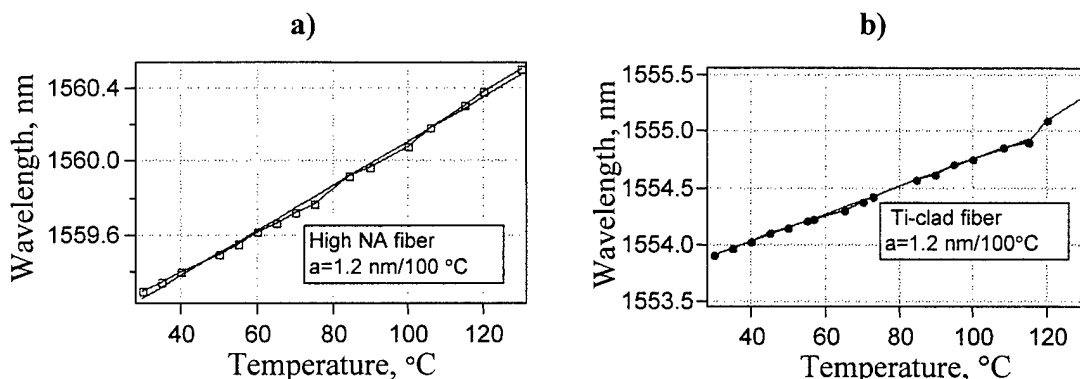


Figure 1. Thermal sensitivity of fiber Bragg gratings in a) high-NA fiber for sensor arrays and b) fiber with Ti-doped cladding having a zero thermal expansion coefficient. The similarity of the slopes (the coefficient labeled “a” in the inset) of the two plots shows that the cladding’s thermal expansion has a negligible effect on the grating’s thermal sensitivity.

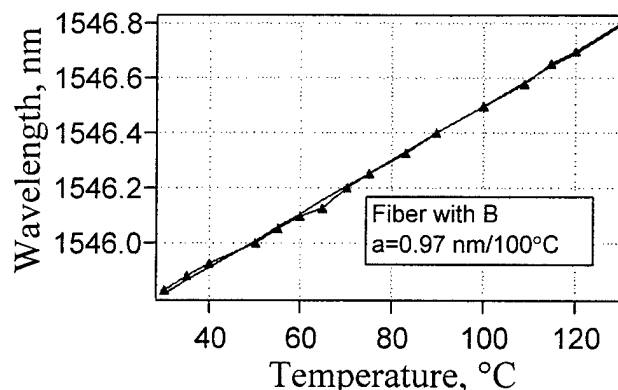


Figure 2. Thermal sensitivity (the “a” coefficient in the inset above) is reduced by ~20% in a fiber with a high concentration of boron.

Figure 1 shows that reducing the thermal expansion of the cladding did not improve the thermal properties of fiber gratings written in the fiber core. However, Fig. 2 shows that in the fiber with B codoping there was a ~20% decrease in the grating’s thermal sensitivity, making gratings written in this fiber thermally more stable and therefore more suitable for locking pump lasers.

2) Grating stability under light exposure

We also tested the stability of the gratings written through the polymer to high-intensity infrared light traveling in the core of the fiber. The gratings under the test are designed for wavelength locking of semiconductor lasers used for pumping erbium-doped fiber amplifiers.

Intense light at 1.06 microns from an ancient but powerful cw Nd:YAG laser (Coherent Antares) was launched into the fiber containing a Bragg grating. The 400 mW of IR power exceeded that available from pump lasers presently on the market. (The typical power of a pump laser at 980 nm is 100-200 mW.) Figure 3 shows that the reflectivity of the fiber grating didn’t change even after 500 J of IR energy. There was a barely resolvable shift in the grating wavelength by 0.1 nm to shorter wavelengths. This shift could be caused by erasure of any unstable, uniform portion of the grating, because this would alter the refractive index and thereby change the resonance wavelength of the grating.

The stability of these gratings to high IR energies makes them suitable for the commercial application of stabilizing the output of high-power semiconductor laser sources.

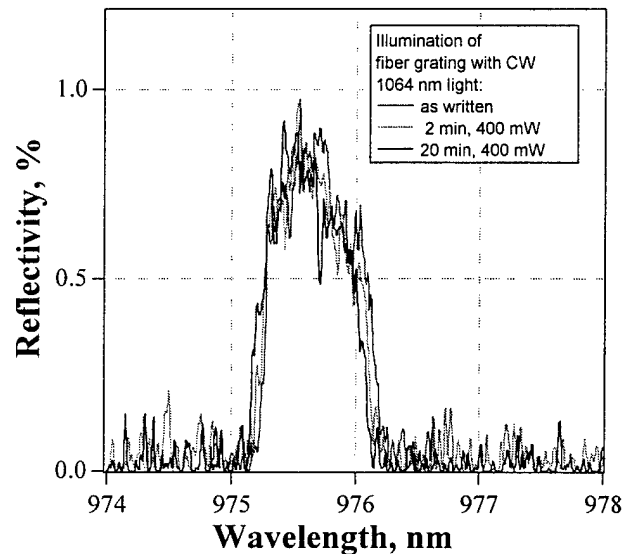


Figure 3. High-intensity infrared exposure of a fiber Bragg grating written through the polymer does not change the grating's properties.

3) Grating reliability under mechanical stress

We also investigated the effect of thermal annealing on the strength of fiber gratings. The polymer-coated fibers were heated for 1 min and then tested on a commercial proof tester manufactured by Vytran.

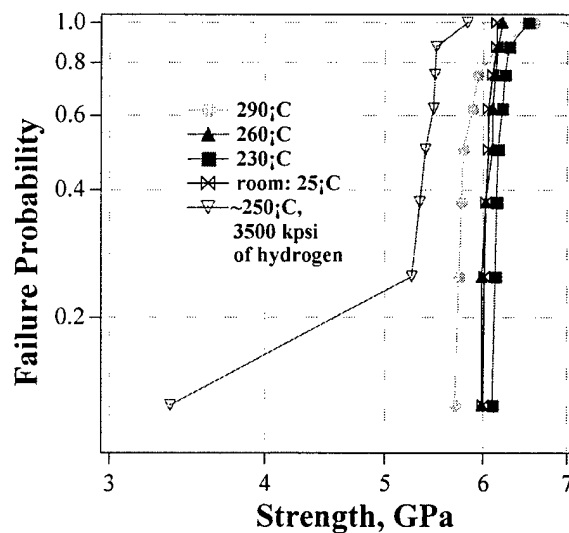


Figure 4. High-temperature annealing (up to 260°C) of ordinary fiber gratings does not degrade their mechanical strength. However high-temperature annealing of hydrogen-loaded fibers does cause a significant degradation of mechanical strength.

Figure 4 shows that heating the fiber to 230°C slightly improved the fiber strength. This was probably due to a decrease in the relative humidity and a drying of the polymer coating. (Water markedly decreases a fiber's strength.) Further heating of the fiber to 290°C caused the fiber's strength to decrease only slightly, possibly due to reactions of the fiber's coating with fiber's silica surface. However, hydrogen-loaded fibers showed a marked decrease of strength after annealing. For this reason we have decided to avoid high-temperature annealing and hydrogen loading of fibers in our grating production facility.

4) Design constraints for Bragg gratings for locking pump lasers

We asked several manufacturers of pump laser to give us their requirements for gratings to stabilize their lasers. Gratings for pump stabilization should have a bandwidth between 0.3 nm and 1 nm, and a reflectivity in the range of 1% to 7%. From the uncertainty principle, for a uniform fiber Bragg grating a bandwidth of 0.5 nm requires a grating length of about 1 mm at 980 nm. However, a 1-mm long grating would have a maximum reflectivity below 0.5% for a grating written through polymer fiber coating. This is because the polymer coating limits the total fluence of UV light that can reach the fiber core, which limits the achievable core index modulation to approximately 10^{-4} .

In order to make fiber gratings with a larger reflectivity we must use a different design. Instead of using very short gratings to produce the required bandwidth, we can use chirped gratings. A chirped grating can produce the required bandwidth and reflectivity. The trade off is that the grating is longer, typically a cm or so. A concern of such long fiber Bragg gratings is their sensitivity to bending. Therefore, we studied the sensitivity of our longer gratings to mechanical bending, and the results are shown in Fig. 5. We were able to bend our gratings down to a 6-mm radius without a noticeable change in the grating spectrum. The telecom requirement is a 16-mm bending radius.

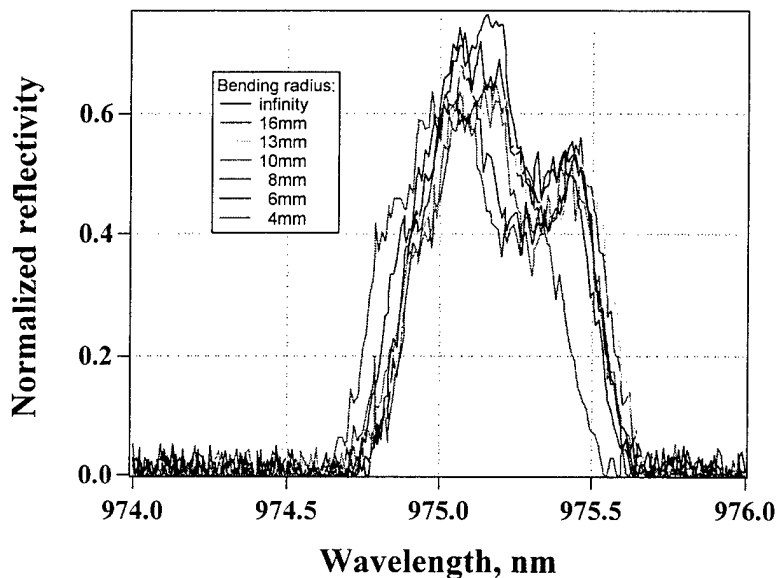


Figure 5. Bending of fiber Bragg gratings written through the polymer coating. The grating resonance is relatively immune to bending down to a 6-mm radius. The grating reflectivity is 2% and its length is 2 cm.

- In our next set of experiments, we fabricated fiber Bragg gratings designed for 1480-nm pump lasers. These gratings required a bandwidth of ~ 2 nm and a reflectivity of $\sim 5\%$. Figure 6 shows an example of a fiber grating for the 1480-nm wavelength range written through fiber's polymer coating. This grating was written in a fiber having a boron co-doped core. The main drawback of B co-doped fibers for 1480-nm applications are their relatively high absorption ($\sim 0.2\text{dB/m}$) in the 1480-nm range). Fibers doped only with Ge to the required numerical aperture (N.A. = 0.13) are not sensitive enough. Further optimization of the fiber composition is required.

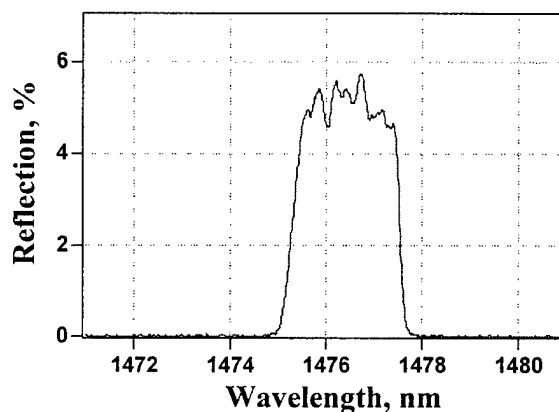


Figure 6. A Bragg-grating resonance in the 1480-nm wavelength range formed in a B-co-doped fiber through the fiber's polymer coating.

Publications:

Results from this STTR project were presented November 6, 2000 at the "Photonics East" conference in Boston Massachusetts. Dr. Dmitry Starodubov, the Principal Investigator of this STTR project, taught a short course entitled, "Fiber Gratings for WDM Applications." Relevant viewgraphs from that course are attached to this report.

Inventions:

NONE

Fiber Gratings for WDM Applications

Dmitry S. Starodubov

D-STAR Technologies, Inc.

Lecture outline

Time

- | | |
|---|------------------------------|
| 1. Basics of fiber photosensitivity | 1.30 pm |
| 2. Fabrication techniques of fiber gratings | |
| 3. Fiber grating operation | |
| 4. Reliability of fiber grating components | Coffee
break
~ 3.30 pm |
| 5. Fiber gratings in WDM systems | |
| 6. Questions | 5 pm |
-

My thanks to Jack Feinberg, Victor Grubsky and Alan Willner

1. Basics of fiber photosensitivity

Photosensitivity physics

Fibers are exposed by UV light to make gratings.

UV light changes refractive index of the fiber core.

Main things to know about photosensitivity of fibers:

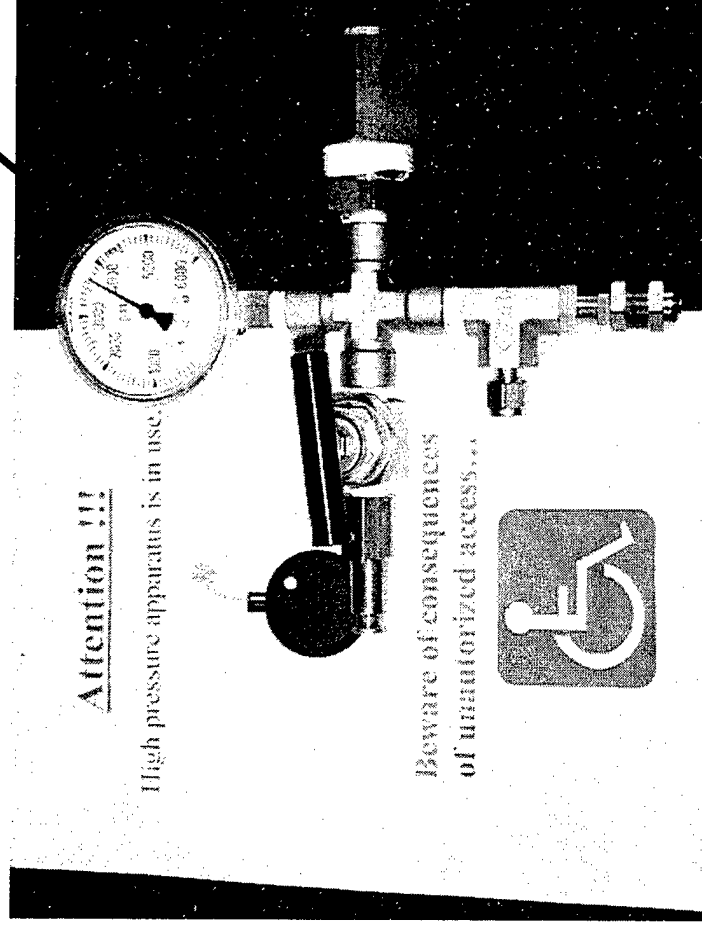
- *What changes the index of glass?*
 - *How big can be the index change?*
 - *Which UV light source should be used for writing?*
 - *What are the main problems?*
-

Two stories in one

1. Fibers without hydrogen

2. Fibers loaded
with hydrogen

4000 psi



Photosensitivity mechanisms summary*

Without hydrogen

Positive index change (Type I) *Negative index change (Type II a)* *Core-cladding interface damage (Type II)*

Index change: 10^{-4} - 10^{-3}

(Nonlinear dependence of index on fluence)

Oxygen deficient defect-related structural transformation of glass

Writing wavelengths: 240 nm, 330 nm, also 193 nm

Introduces birefringence when writing with polarization across the fiber

Hydrogen loaded

Positive index change, OH group formation

Index change: up to 10^{-2}

(Linear dependence of index on fluence)

Photochemical reaction of hydrogen with Ge-O bonds

Writing wavelength: <310 nm (the shorter the better)

Requires hydrogen outgassing which changes index by $\sim 10^{-3}$

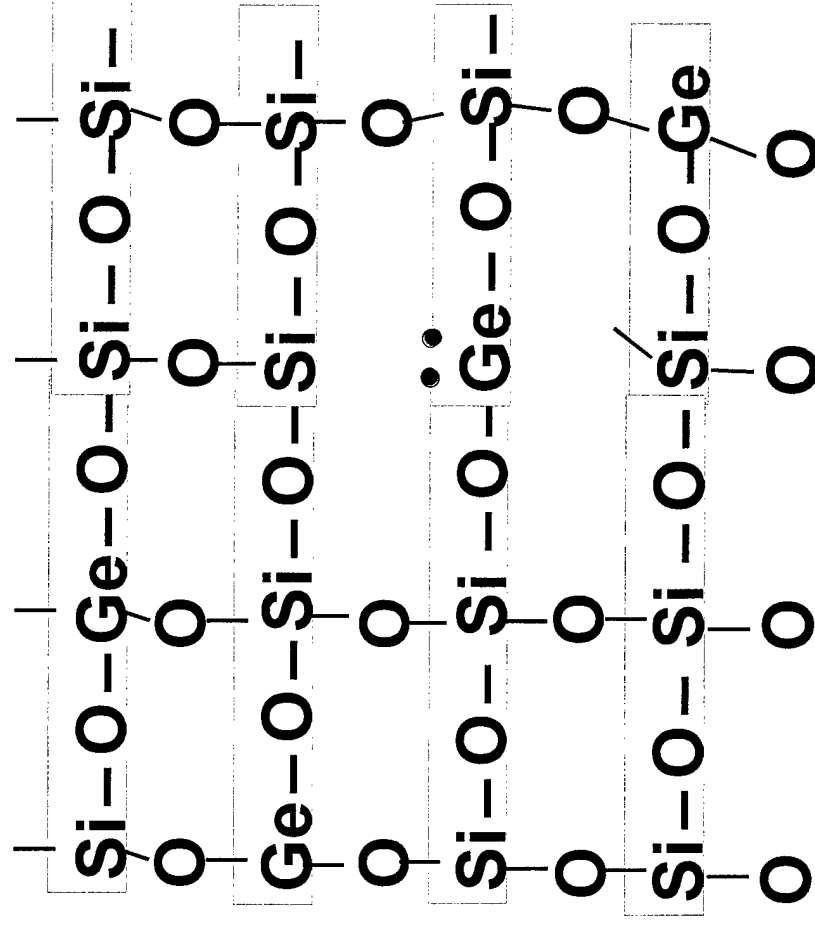
*** this is ALL YOU HAVE TO KNOW!**

Photosensitivity of glass (no hydrogen)

- Pure silica has very little photosensitivity.
- Ge is in the core to raise the core's index, but it also makes the core photosensitive*.
- It is the Ge *defects* that matter (if there is no hydrogen present).
- Fiber should be made in oxygen-deficient conditions.

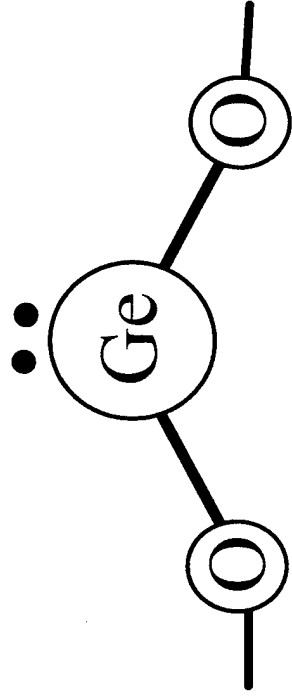
*Less common dopants such as Sn or P could also be used for fiber gratings .

Germanium oxygen deficient defect in silica glass SiO_2

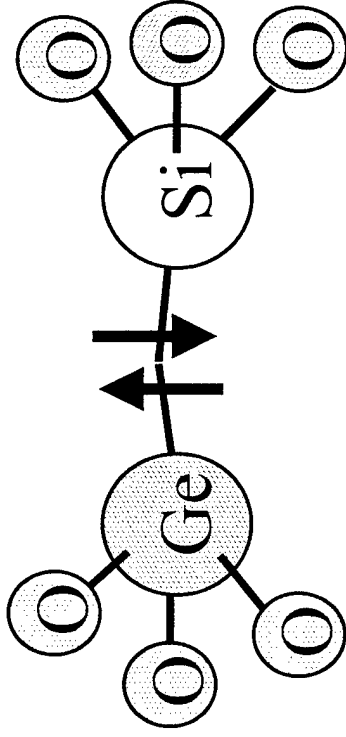


Defect is formed by one Ge atom in 1000.

Models of photosensitive Ge defects



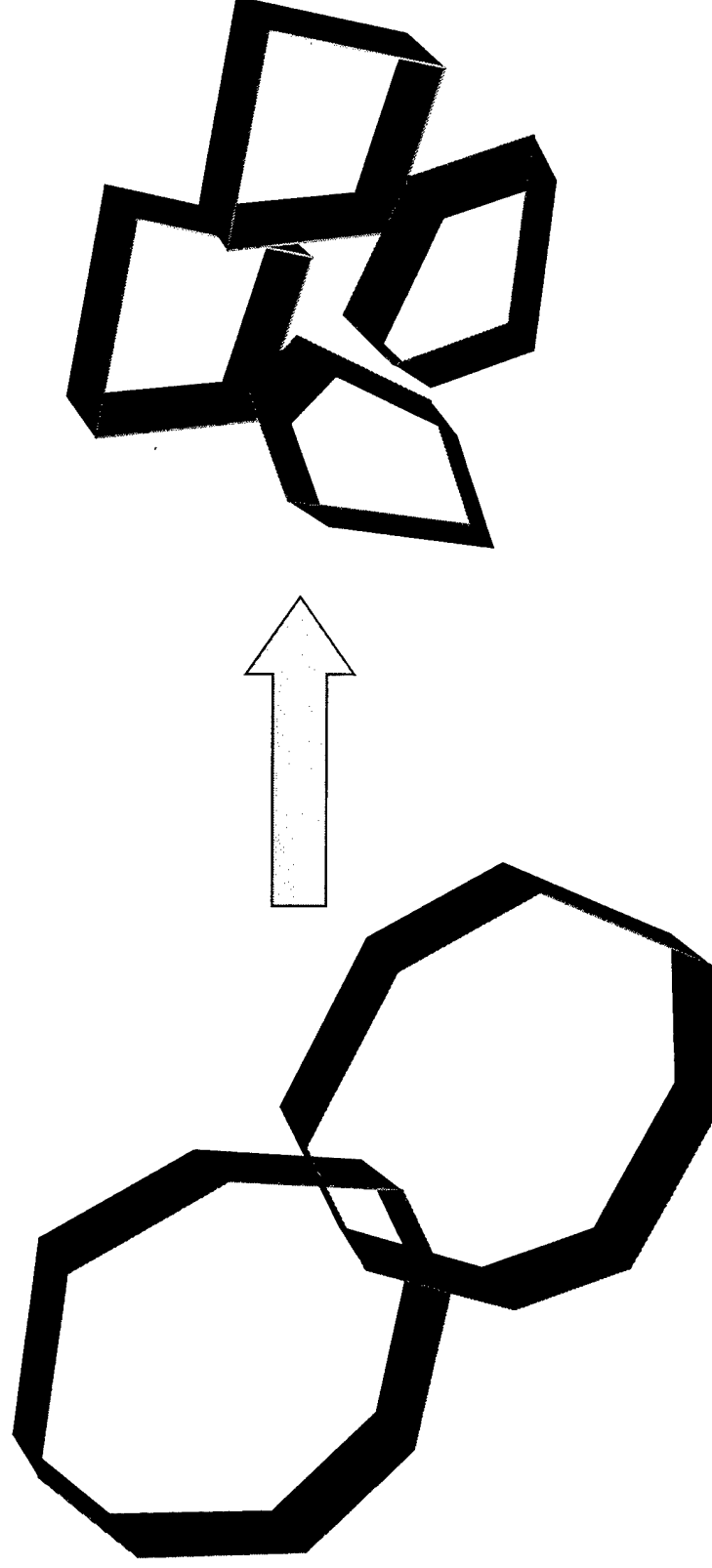
**Twofold
coordinated Ge**



**Oxygen Monovacancy
(wrong bond)**

Oxygen deficiency: Ge atoms have fewer bonds to oxygen

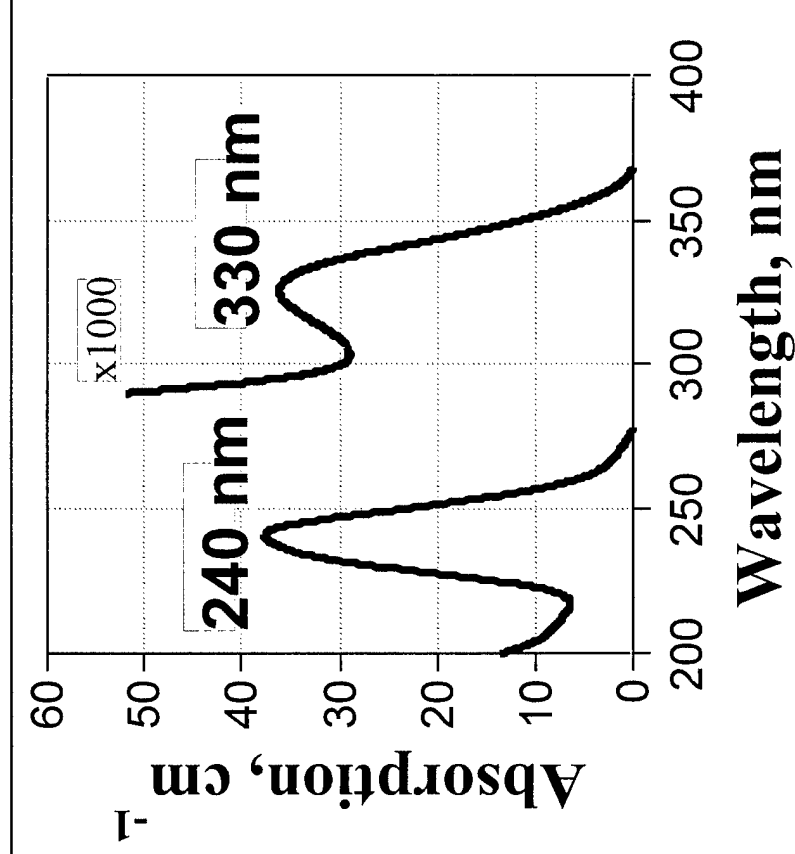
Compaction by ring collapse



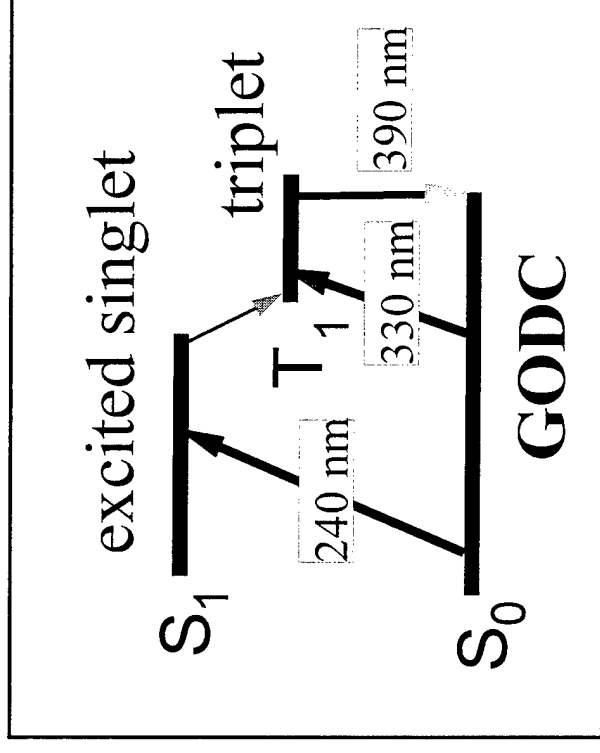
N. M. Lawandy, Opt. Commun. 74, 180-184 (1989)

UV absorption of fiber : Ge defects

Absorption spectrum

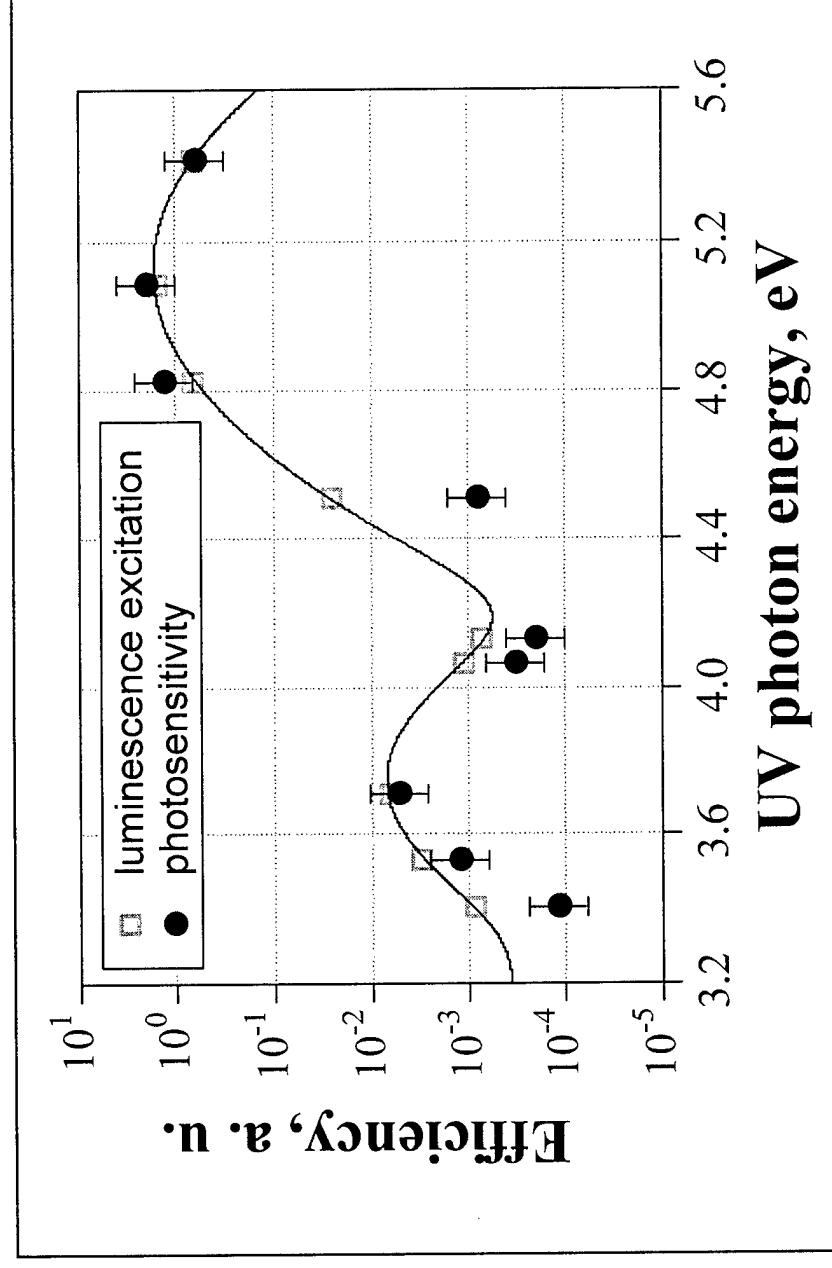


Level structure



GODC stands for Germanium oxygen-deficient center

Spectral dependence, no hydrogen

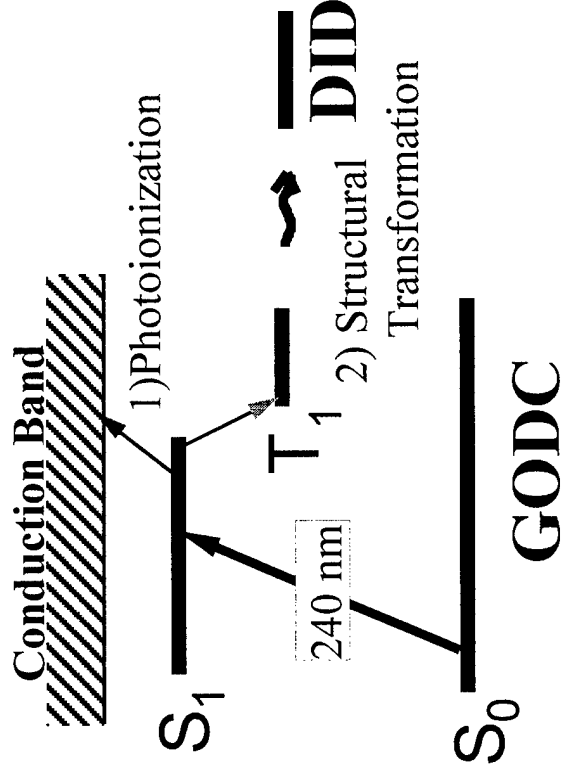


Photosensitivity follows triplet state excitation

Two ways to write gratings

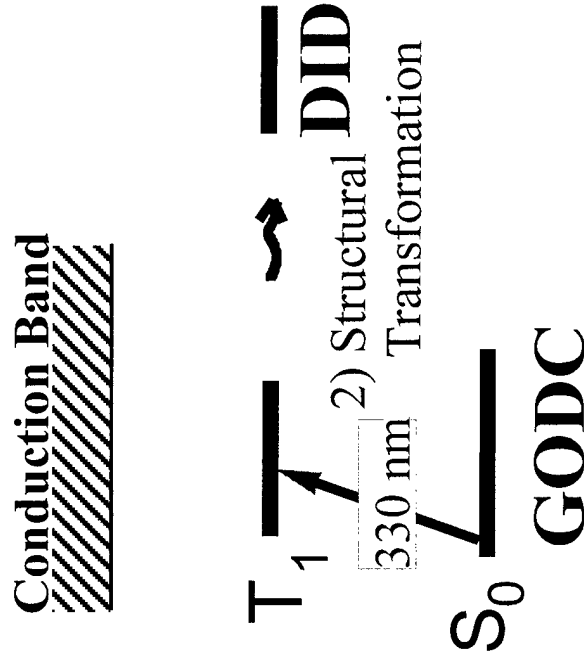
Conventional

Mid-UV 240 nm Writing



Near-UV method

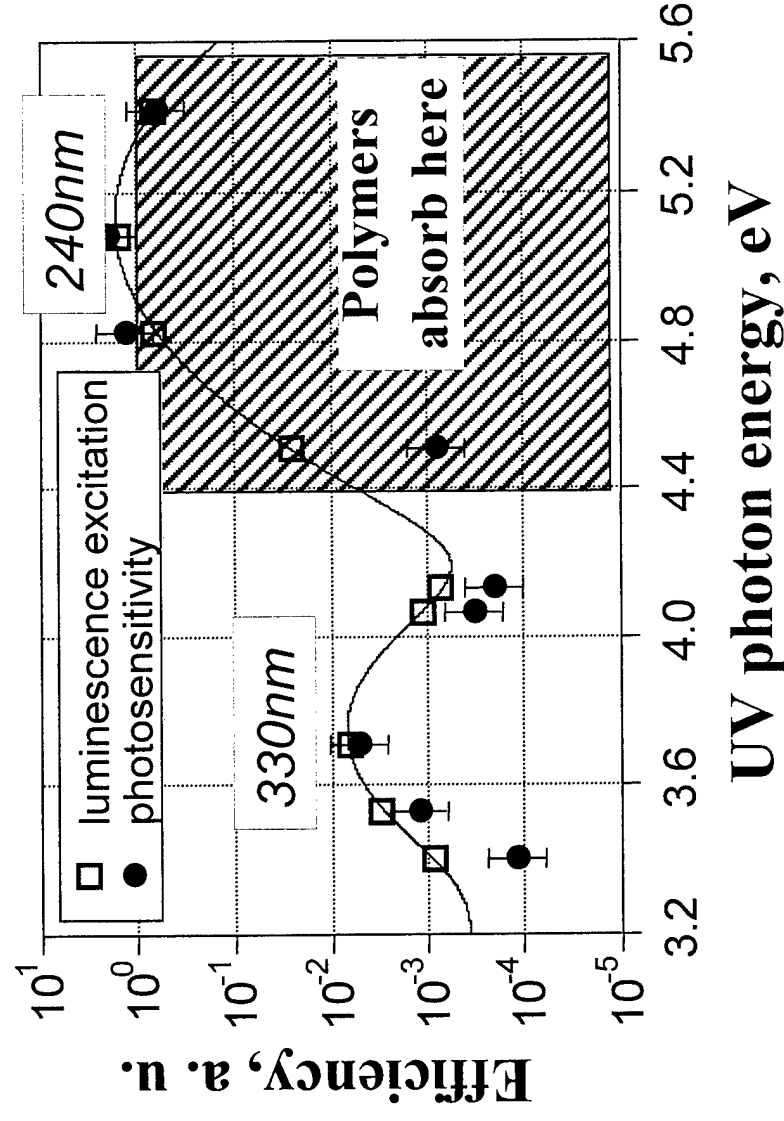
Near-UV 330 nm Writing



Structural transformation changes index.

DID is acronym for Drawing-induced defect.

Writing through polymer: near-UV window

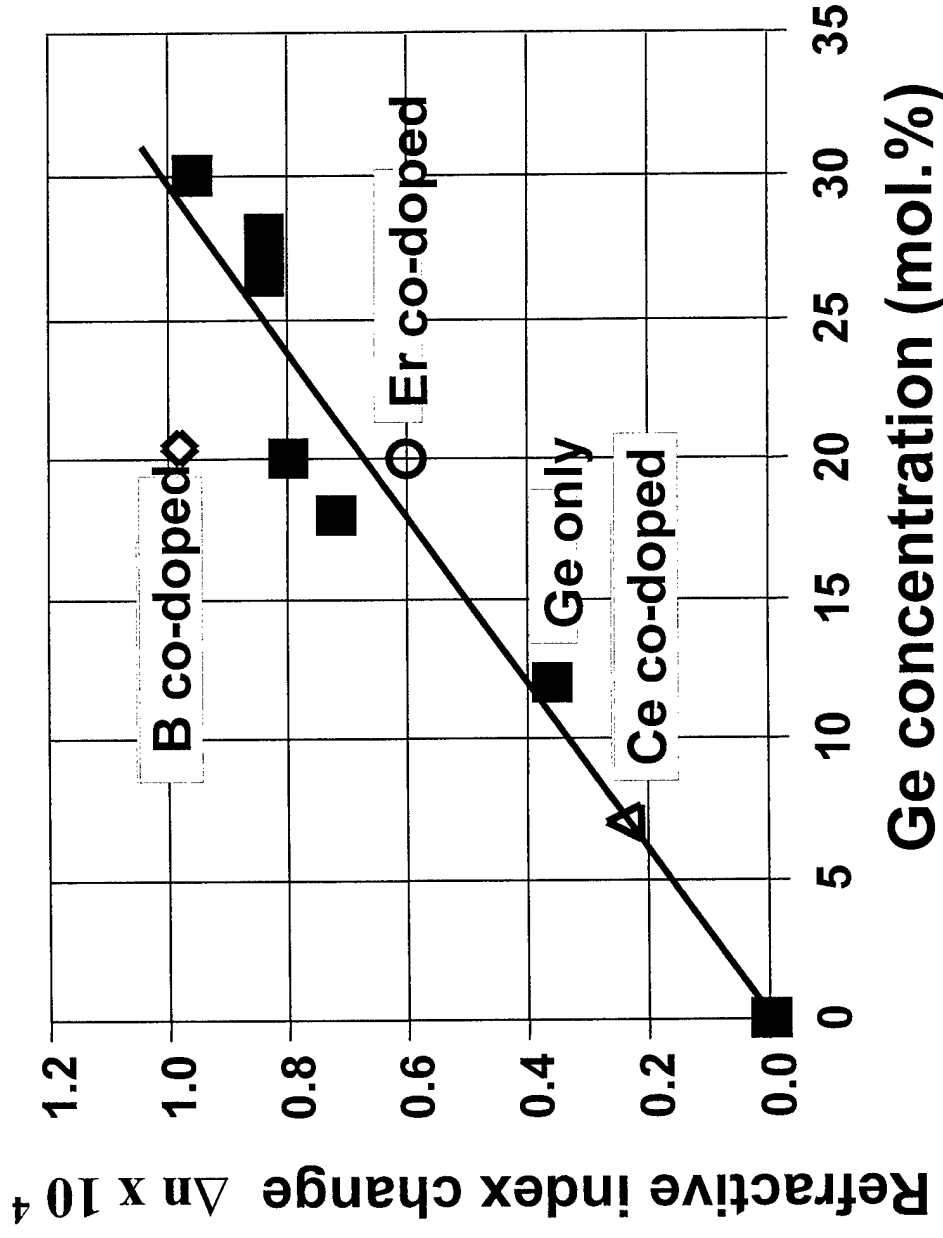


Standard acrylic coatings are transparent to 330 nm light

Near-UV: advantages and drawbacks

- Near-UV argon lasers have excellent beam quality, stability and coherence. No doubling crystal.
 - Less fiber damage than with excimer lasers.
 - Polymer coatings are transparent.
 - Direct triplet transition has weak absorption.
-

Effect of doping on index change



Index change grows linearly with Ge concentration

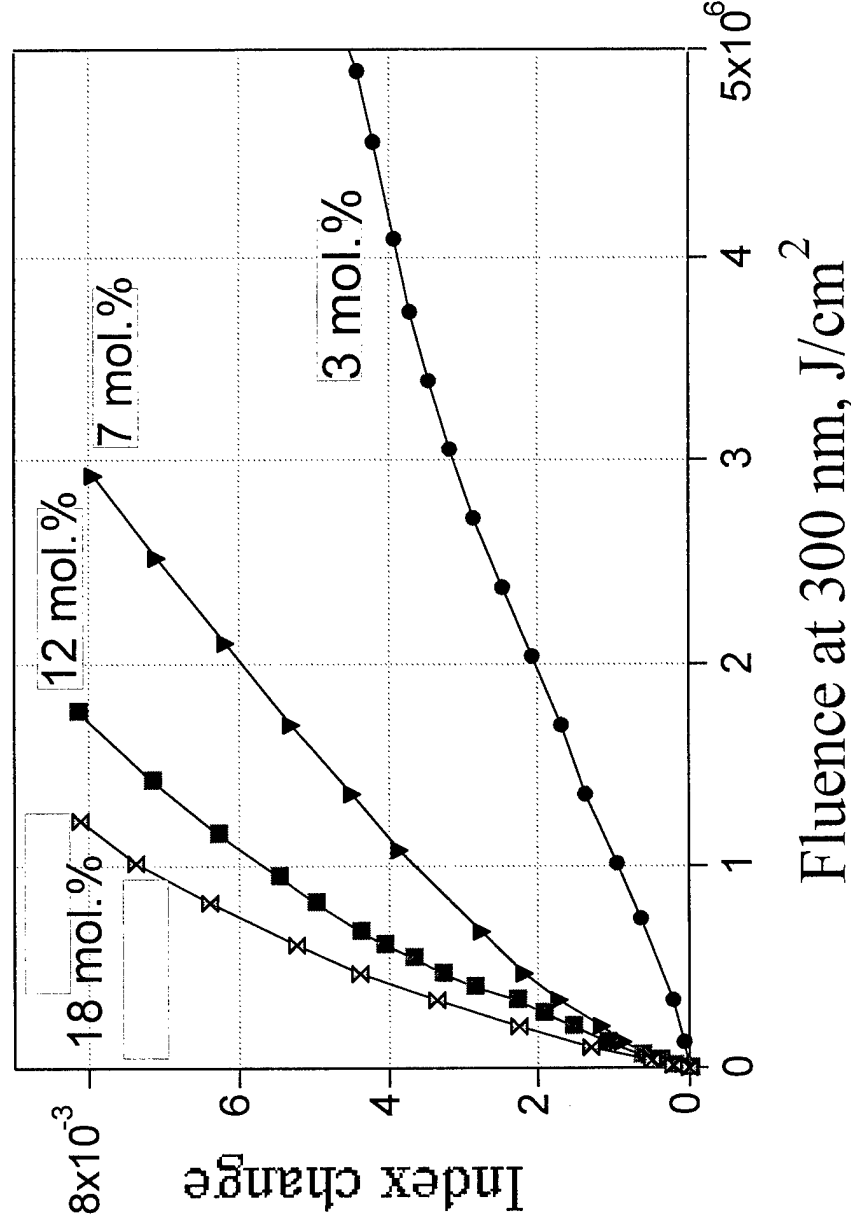
Gratings (without hydrogen): summary of the mechanism

- UV light excites the triplet state of germanium oxygen-deficient centers.
 - Bonds are rearranged. The glass compacts.
 - A local UV induced index change is created.
-

Hydrogen loading of fibers

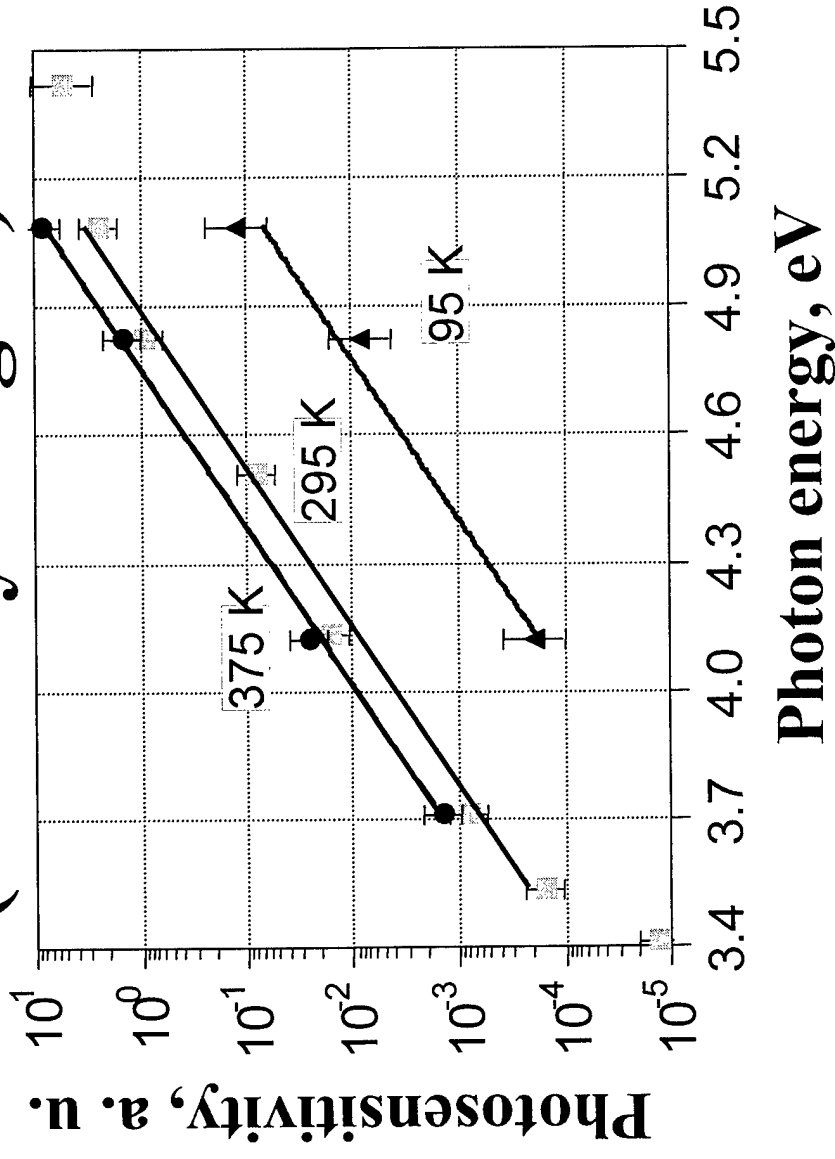
- Fibers are loaded in a chamber with hydrogen or deuterium at $\sim 100\text{-}300$ atm ($1000 - 4000$ psi).
 - Loading time is ~ 2 weeks at room temperature and ~ 1 day at 100°C
 - Indiffused hydrogen increases UV induced index change
 - Post-fabrication annealing is required to outdiffuse remaining hydrogen
-

Germanium doping is essential for photosensitivity in H₂-loaded fibers



In pure silica fiber (no Ge), no Δn could be induced.

Spectral dependence of index change (with hydrogen)



Photosensitivity increases near conduction band edge.

Grubsky et al, Opt. Lett. **24**, 729 (1999)

Index change in hydrogen-loaded fibers

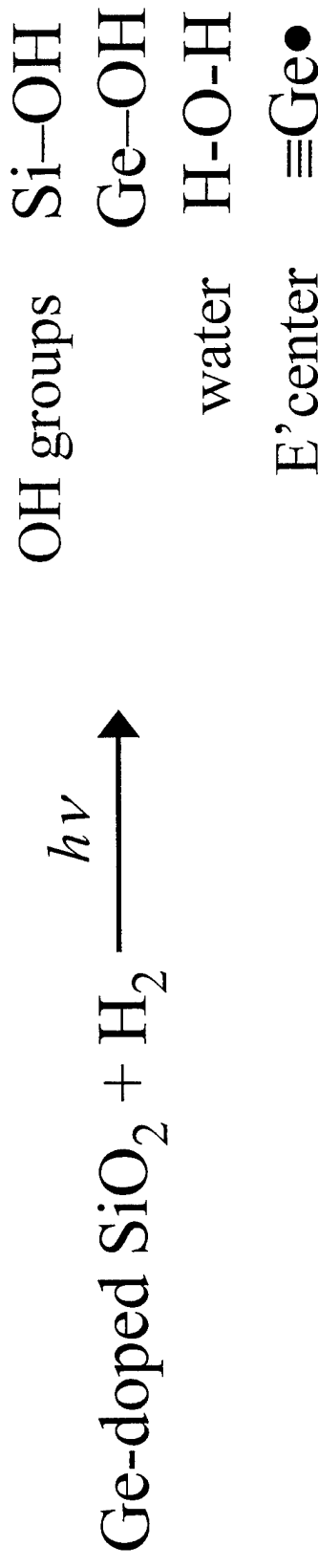
(Lemaire et al., *Electron. Lett.*, 1993)

Hydrogen-free fiber: only Ge defects ($\sim 10^{-3}$ of Ge atoms) contribute.

$$\Delta n_{\max} \sim 5 \times 10^{-4} \text{ (small)}$$

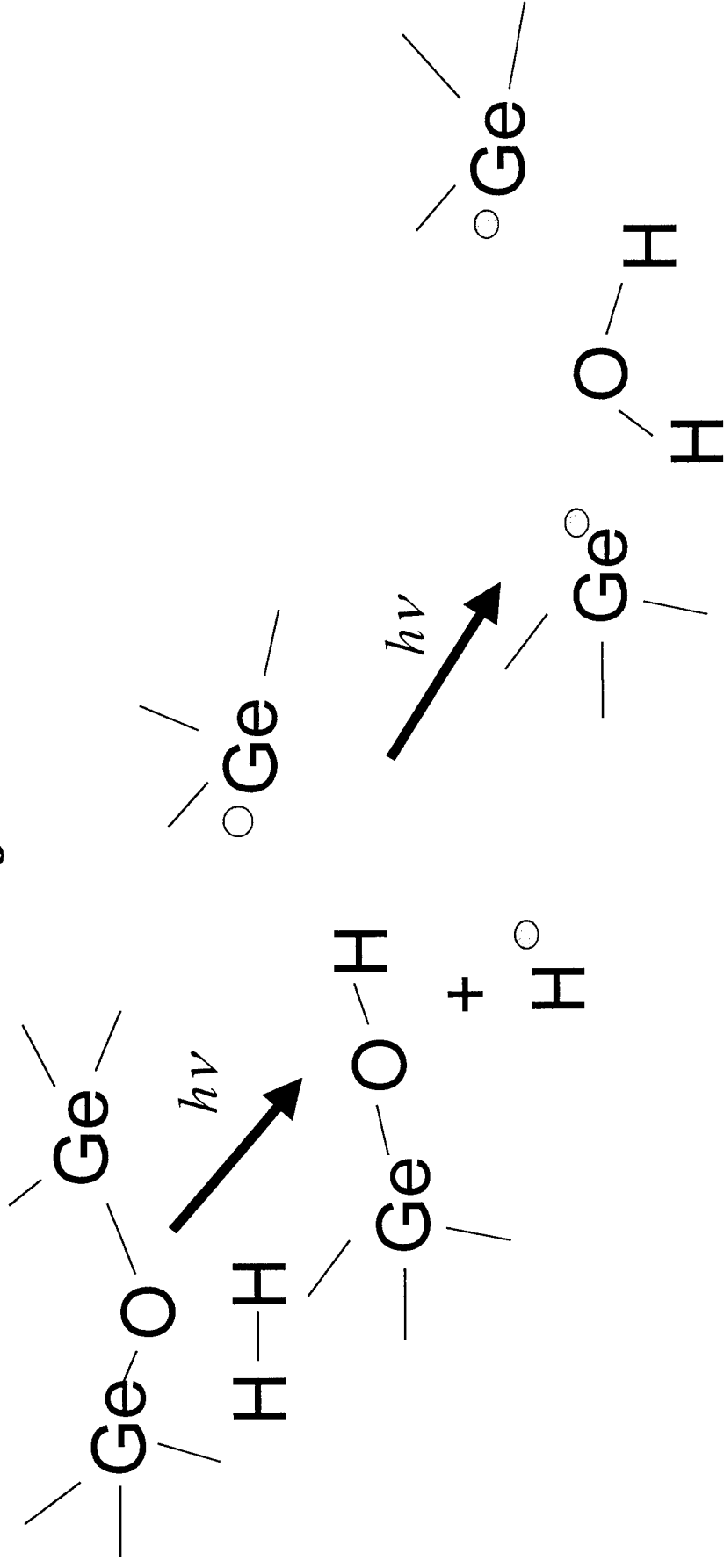
In hydrogen-loaded fibers every Ge contributes.

$$\Delta n_{\max} \sim 100 \times 10^{-4} \text{ (big)}$$



Water formation: Cordier et al., *J. Non-Cryst. Solids*, 1998

Water can be produced by breaking two adjacent Ge-O bonds



Water is one of the products of UV exposure of hydrogen loaded fibers

Grubsky et al, OFC 1999, paper ThD2

Induced OH bonds absorb in infrared spectral region

Absorption band

Absorption
(1 mol. % OH)

Fundamental vibrational
absorption ($\lambda \sim 2.7 \mu\text{m}$)

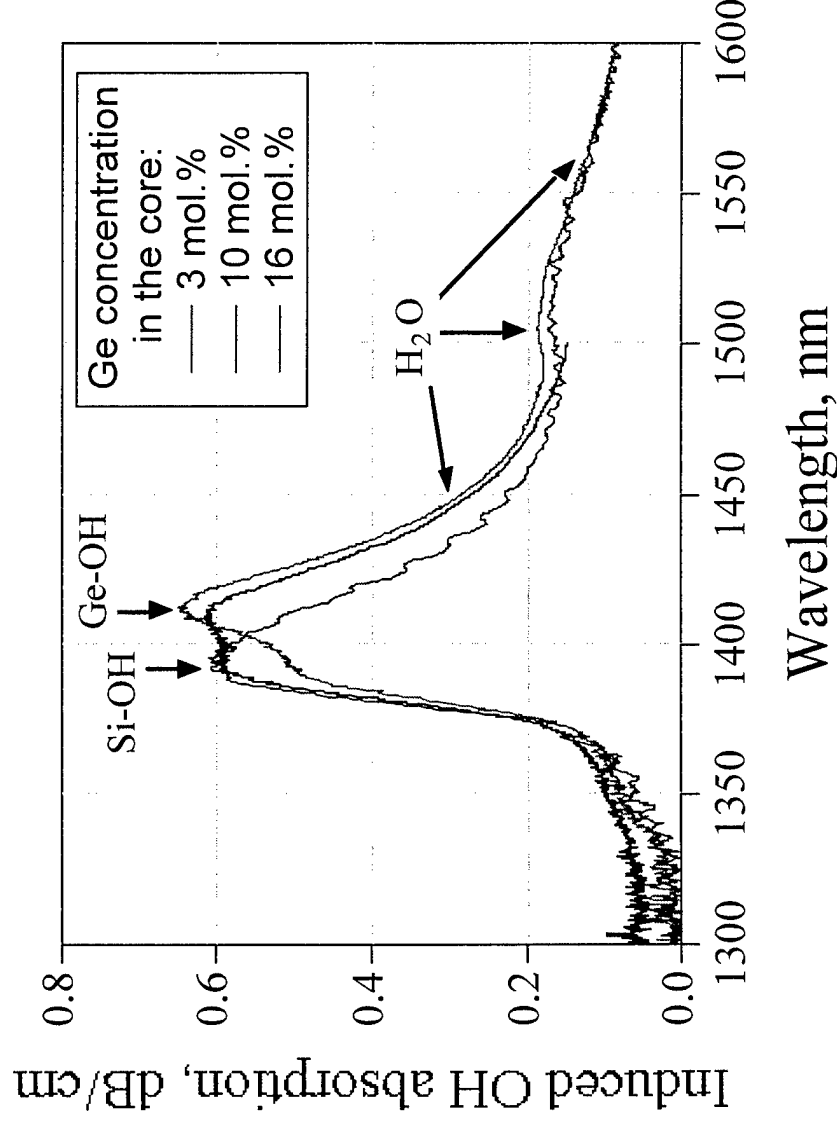
130 dB/cm

Overtone absorption
($\lambda \sim 1.4 \mu\text{m}$)

0.8 dB/cm

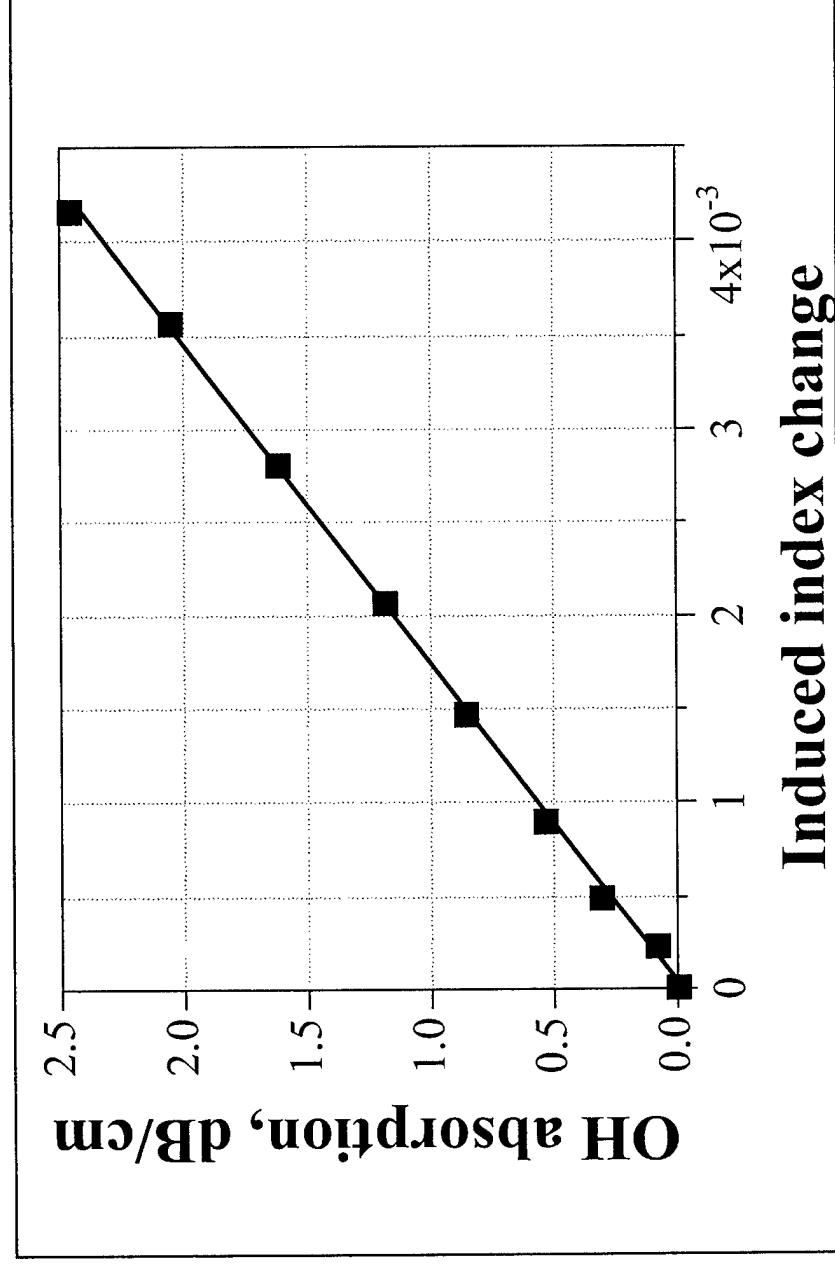
Note: Deuterium-O overtone absorption at $\sim 2 \mu\text{m}$ is away from telecom bands.

UV irradiation with H₂ induces strong absorption due to OH and water



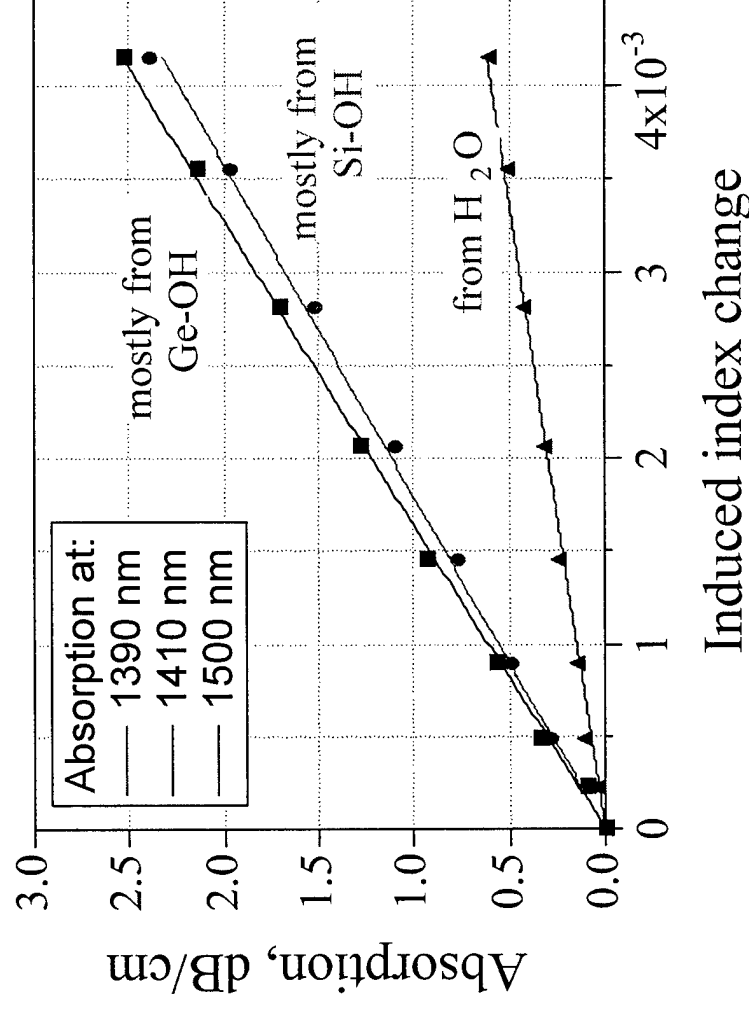
$\Delta n = 10^{-3}$ in all three fibers

OH formation scales with Δn



Regular Ge-O bonds react with hydrogen to form OH

Water as well as UV-induced OH correlate linearly with the index change Δn :

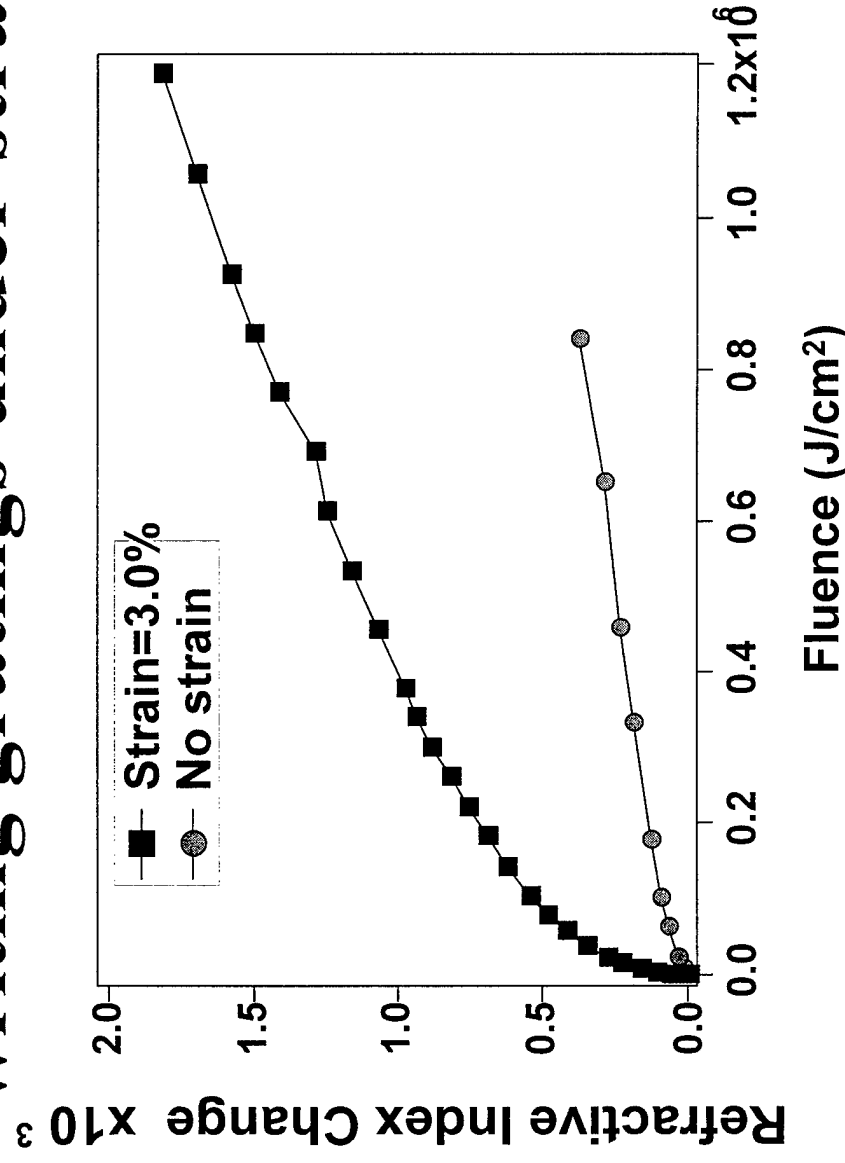


Water is formed even at $\Delta n < 10^{-3}$ and makes gratings less stable

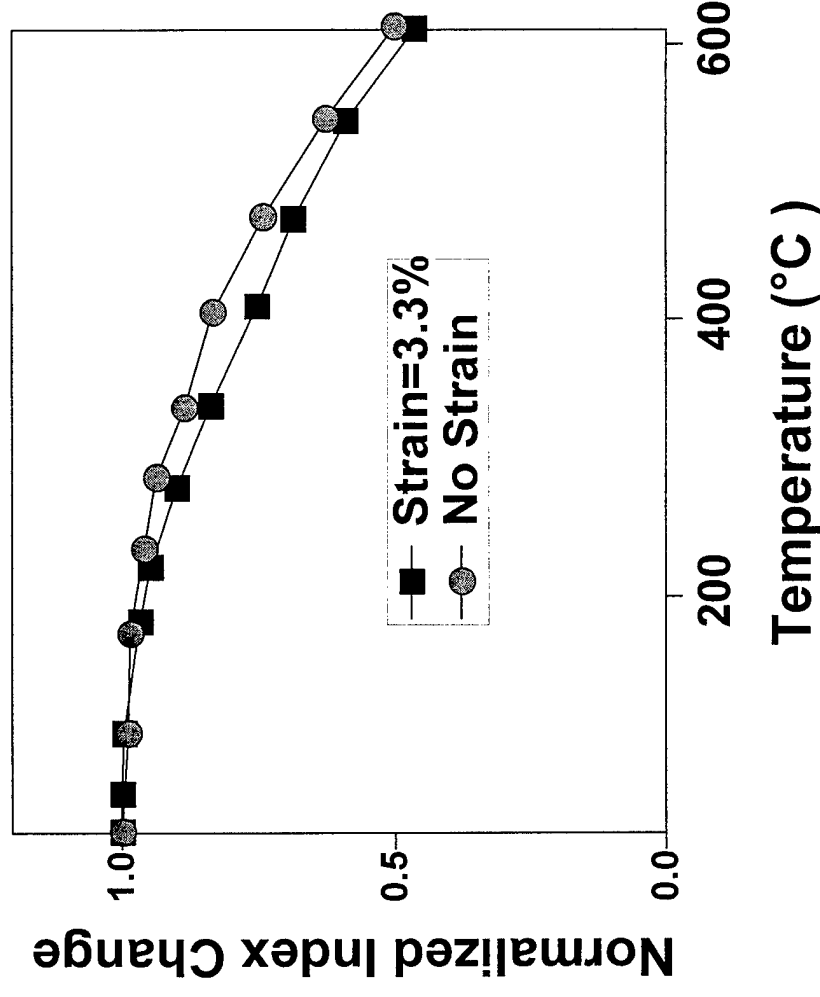
**Is there any hope to get large
index change without hydrogen
loading?**

Yes...

Alternative to hydrogen loading: writing gratings under strain



Gratings written under strain has the same thermal stability as ordinary



No water was created in fiber by stretching so far

Conclusions :

- Index change in fibers without hydrogen is due to bleaching of defects and structural transformation of glass. The index change, however, is too small for some applications.
 - It is possible to write fiber gratings using near-UV light (330 nm). Writing through standard coatings is possible.
 - In hydrogen-loaded glass the index change is due to reaction of hydrogen with regular Ge atoms. OH bonds are formed.
 - Loading step, additional loss, unstable products make hydrogen loaded gratings less attractive for fabrication.
 - Deuterium loading reduces the problem of induced loss.
-

4. Reliability of fiber grating components

Reliability of Fiber Gratings: Summary

-Mechanical reliability

Gratings can break. Mechanical strength is very important for gratings that will operate under strain. Any tiny scratches can dramatically degrade the fiber strength.

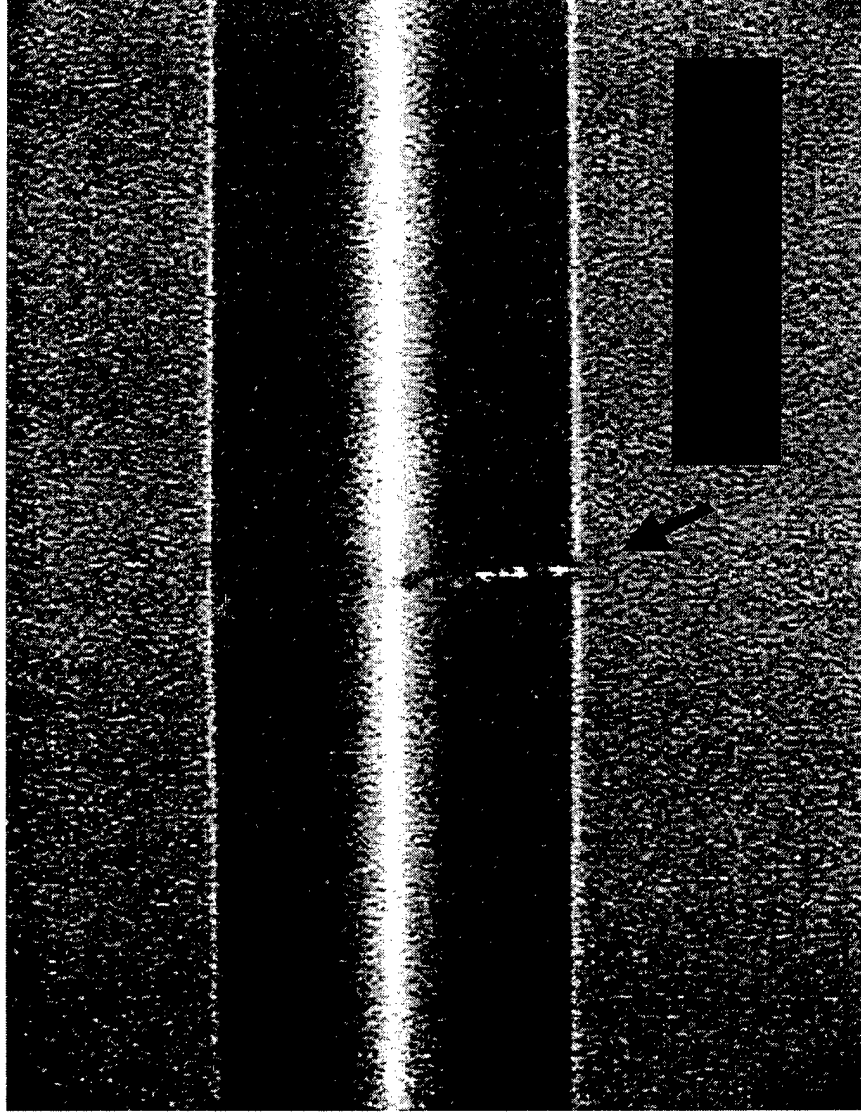
-Stability of optical properties

Gratings decay. Annealing removes unstable portion of the grating and makes the remaining grating more stable.

The grating peak shifts due to the uniform index change.

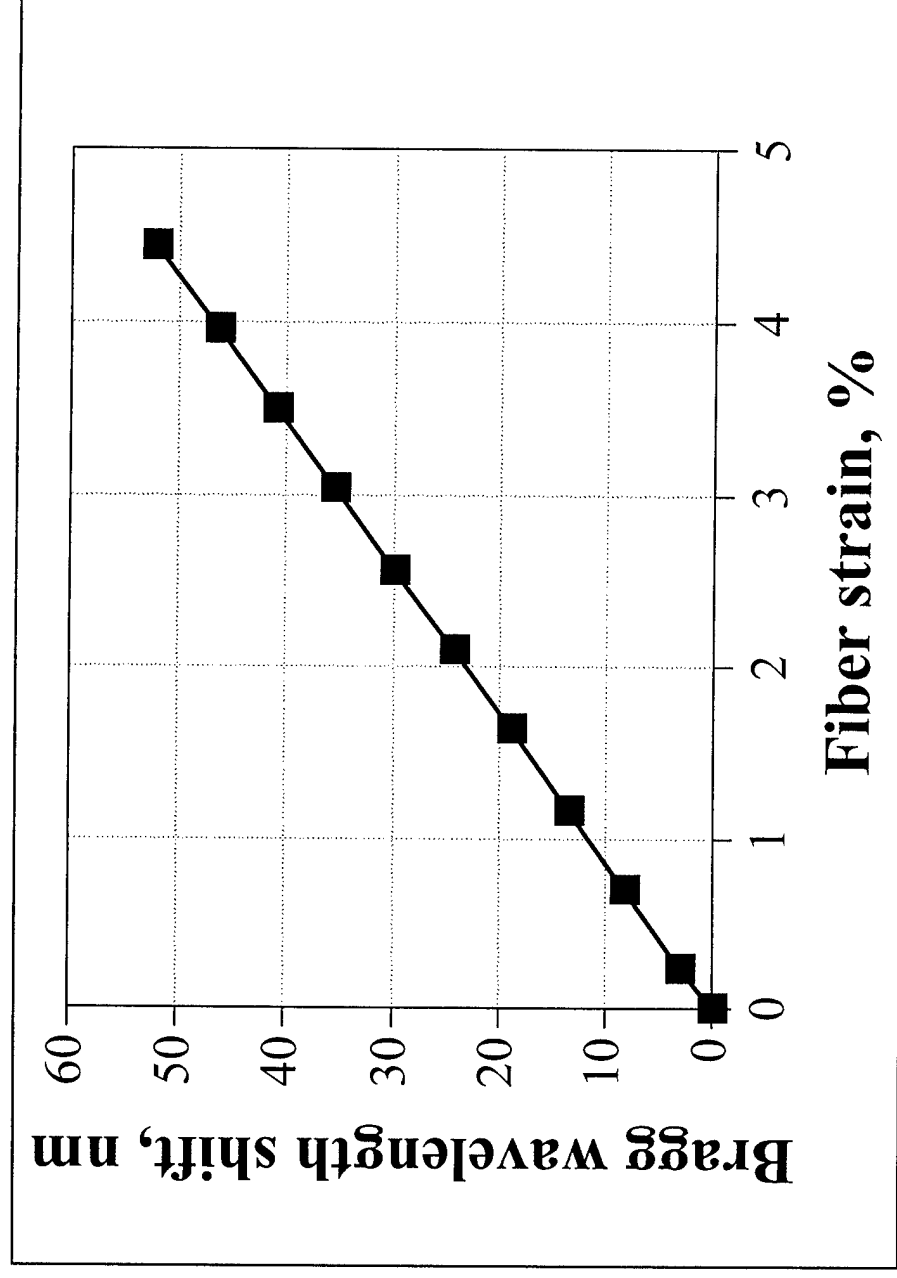
The decrease of index modulation decreases the resonance amplitude.

Reliability is important



Extensive damage of fiber by pulsed UV light: breaking strength is 0.16 GPa (23.1 kpsi)

Tunability: strong grating



Written-through-polymer grating survives >58 nm of tuning by stretching.

Reasons why grating is weaker than fiber

1) “Touching”

Mechanical damage of cladding during stripping, recoating, or handling

2) Effect of UV exposure

Core and cladding can be damaged by UV exposure.

3) Chemical degradation

Processing chemicals and water vapor reduce the fiber strength.

4) Degradation by heat

Combination of chemical degradation and thermal stresses.

Typical grating fabrication procedure

- 1) Strip the fiber coating ["touching"]
↓
 - 2) Write a grating [UV damage]
↓
 - 3) Thermal annealing [thermal damage]
↓
 - 4) Recoat the fiber ["touching"]
-

Weibull plot basics

- Approximately $N=20$ fiber samples are broken.
- The breaking stress is measured for each sample.
- Stress data are sorted from small to big.
- Probability F of breaking is assigned to each stress value: $F=K/(n+1)$, where K is the number of stress value in series

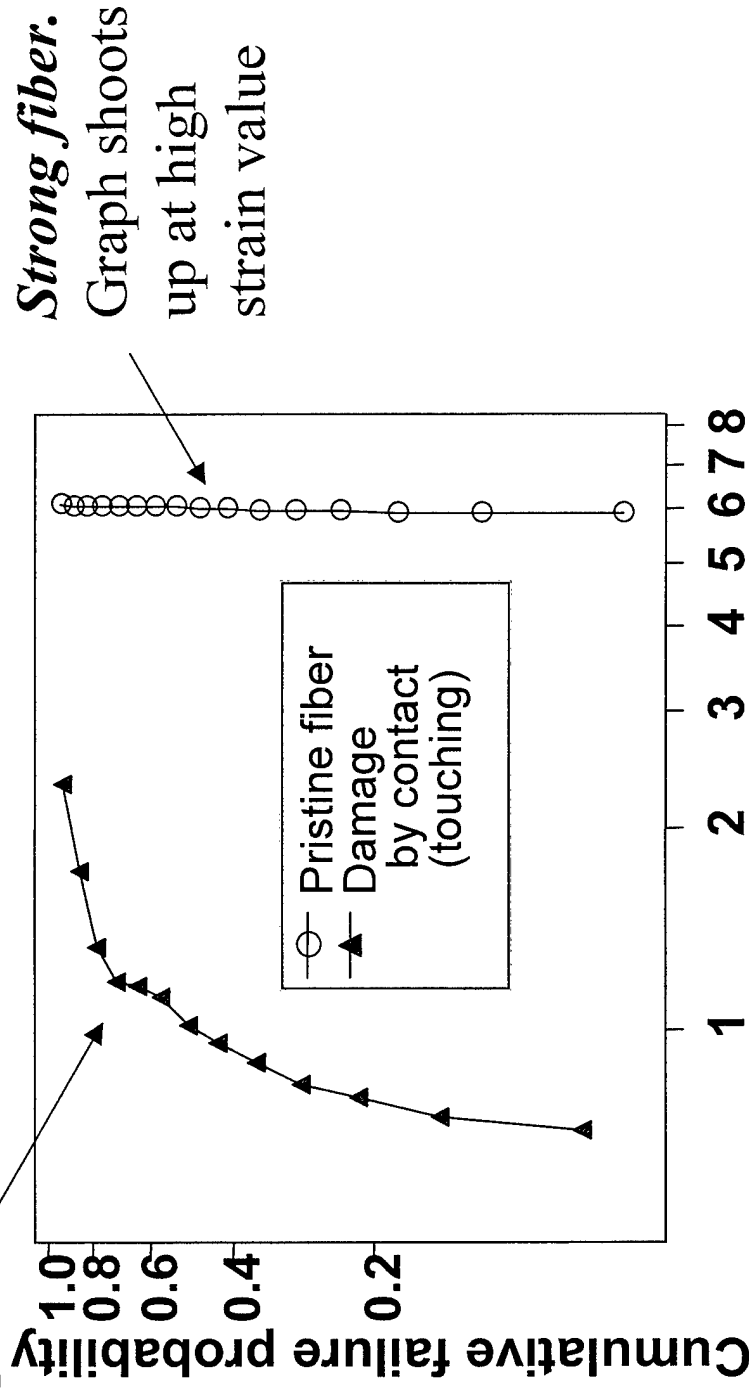
For example, probabilities for 20 samples will be: 1/21, 2/21, 3/21 etc.

- Finally, the result is plotted as log (probability) versus log (stress).
-

Example of Weibull plot: bare fiber handling

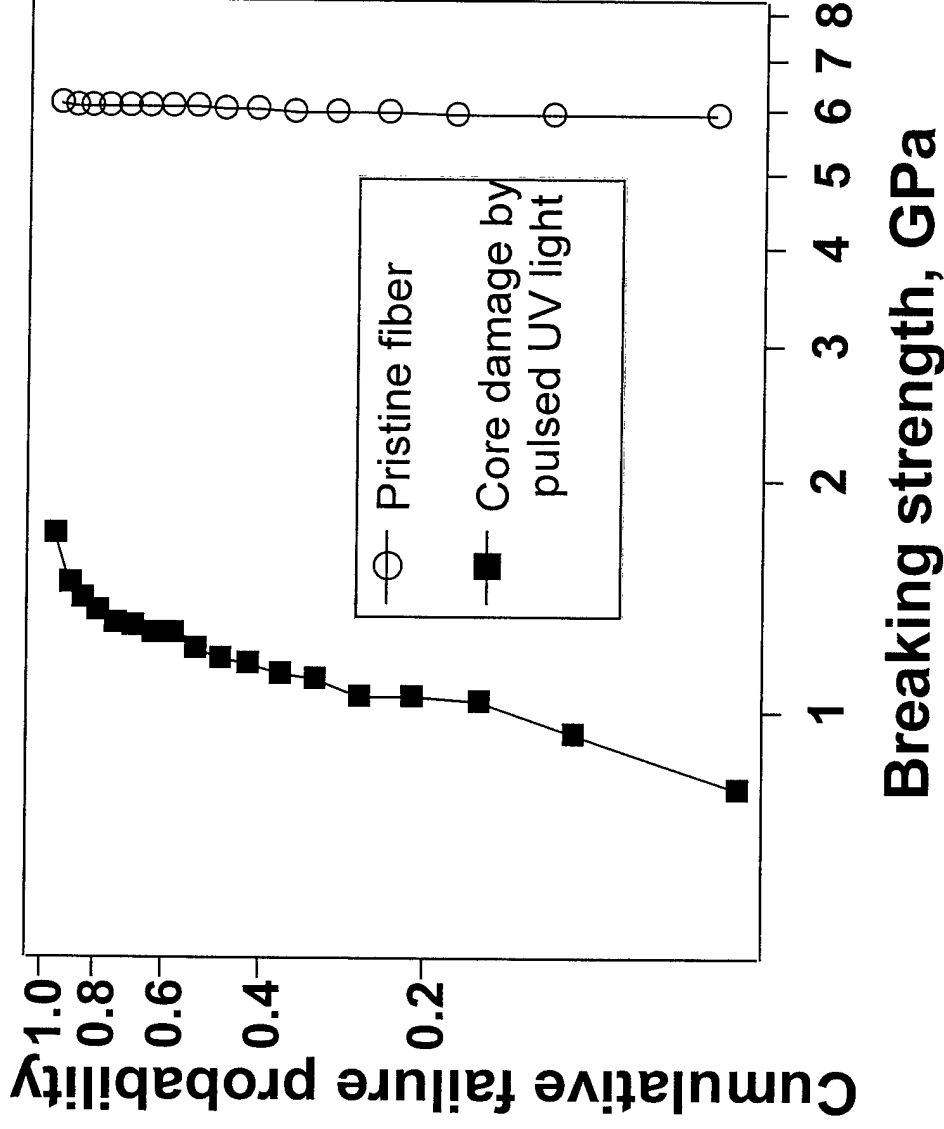
Weak fiber.

Graph is spread
across low strain
values

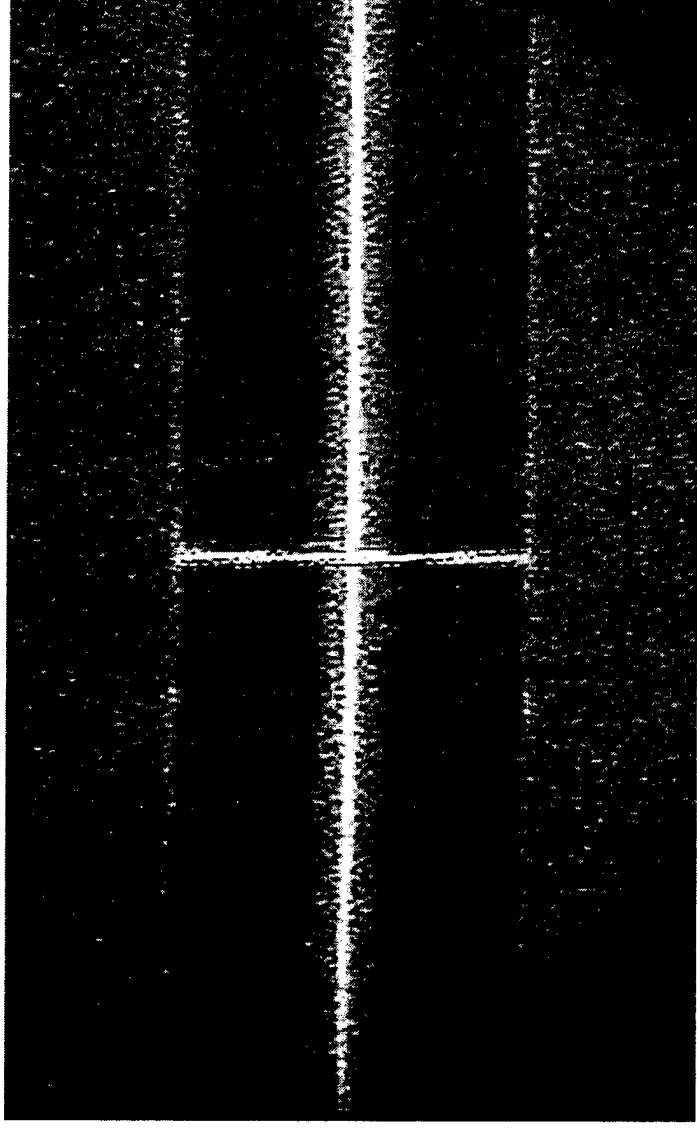


Breaking strength, GPa ← 1 GPa = 149.9 kpsi

Core damage by pulsed UV light

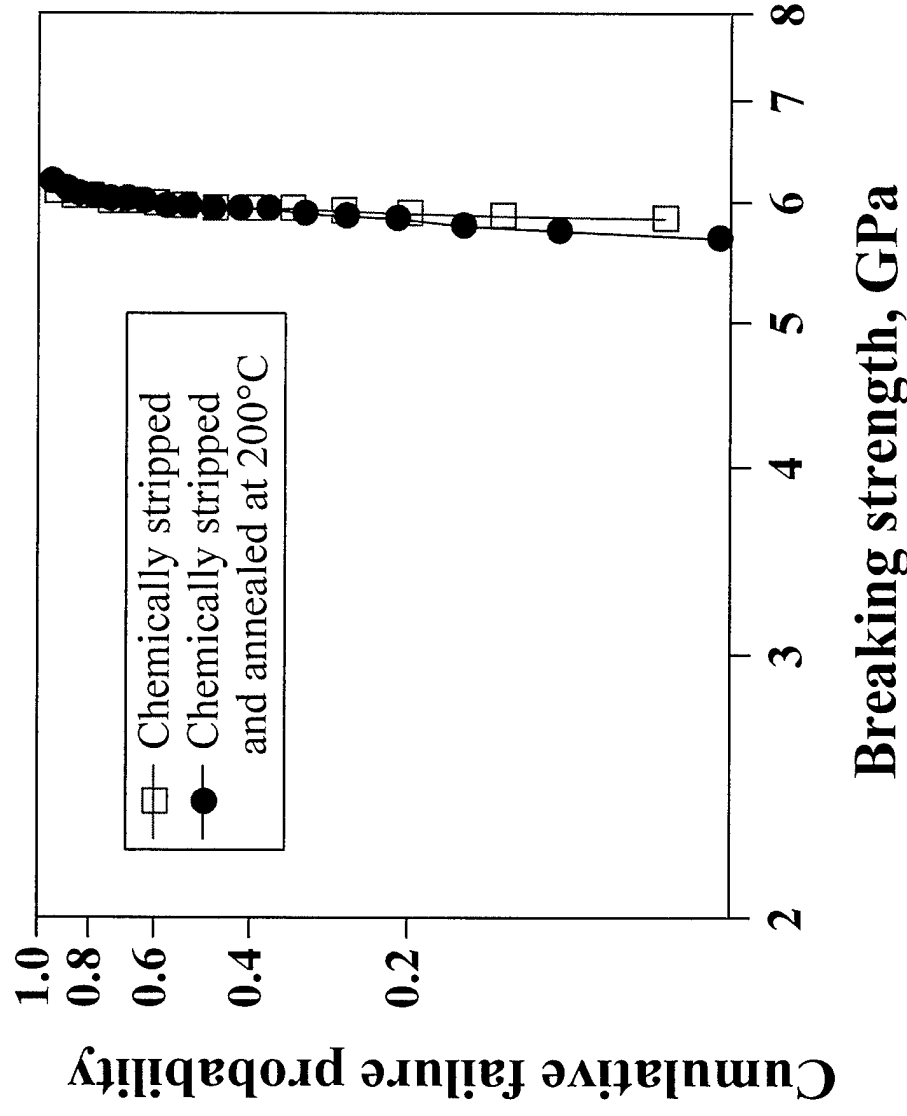


240 nm CW UV light breaks bonds in the cladding



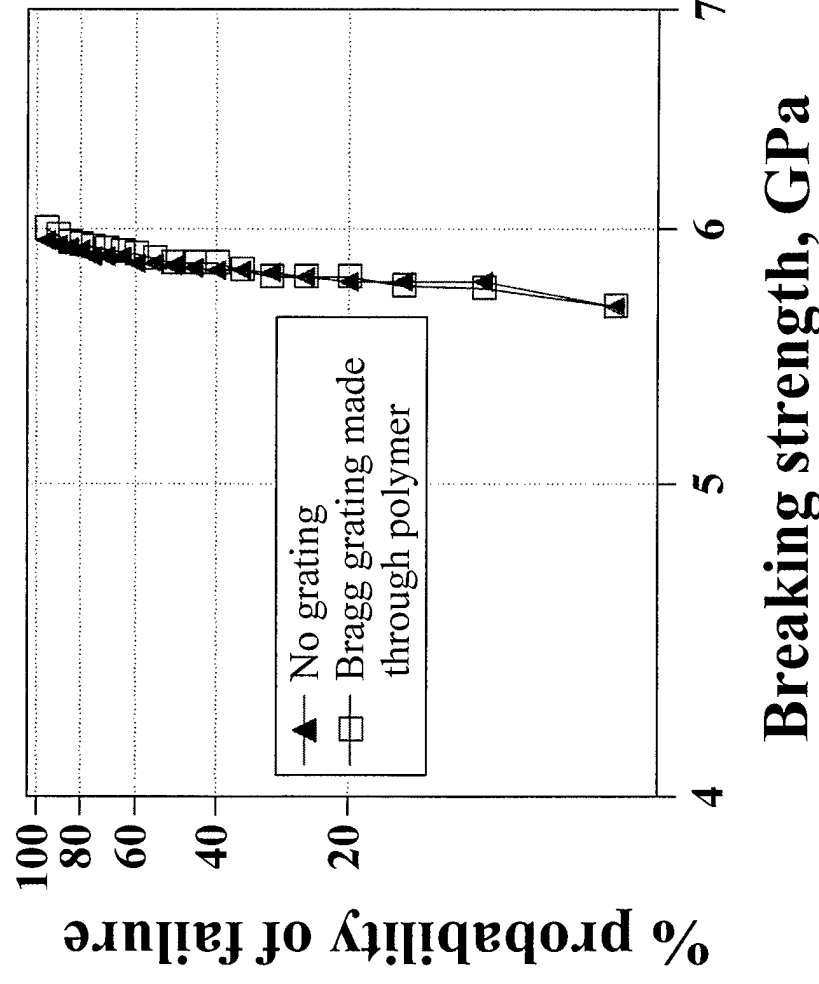
Red luminescence is due to non-bridging oxygen, fiber is still strong

Thermal annealing for 2 min. at 200°C



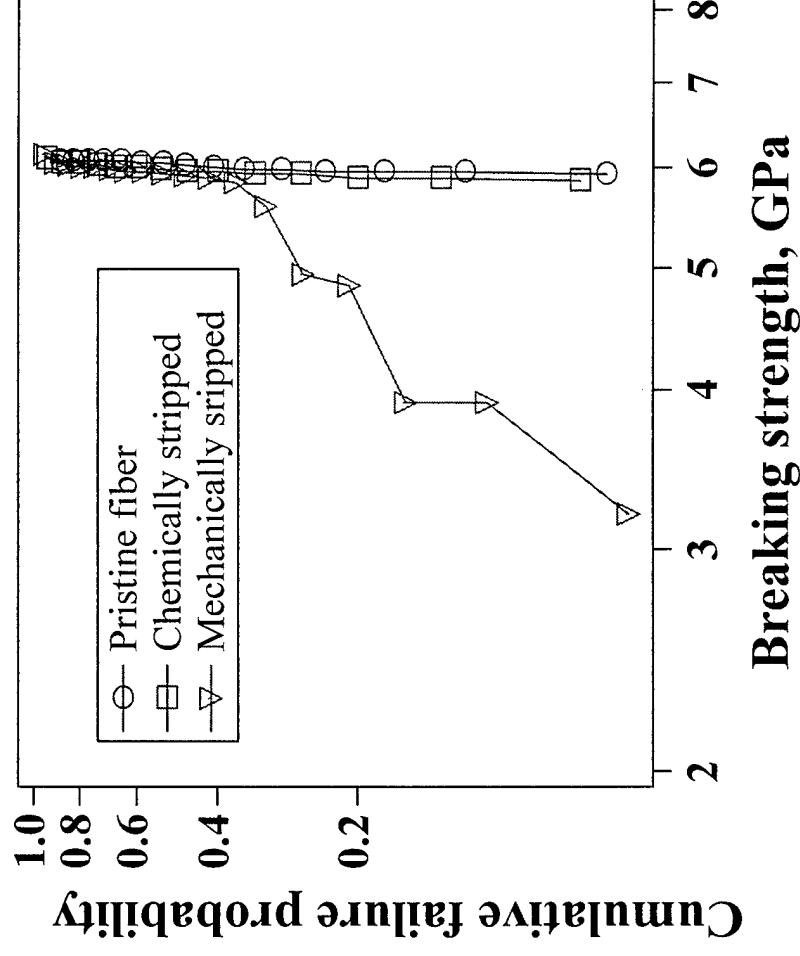
Thermal annealing does not reduce fiber strength

2dB Bragg gratings written through the polymer coating, no hydrogen



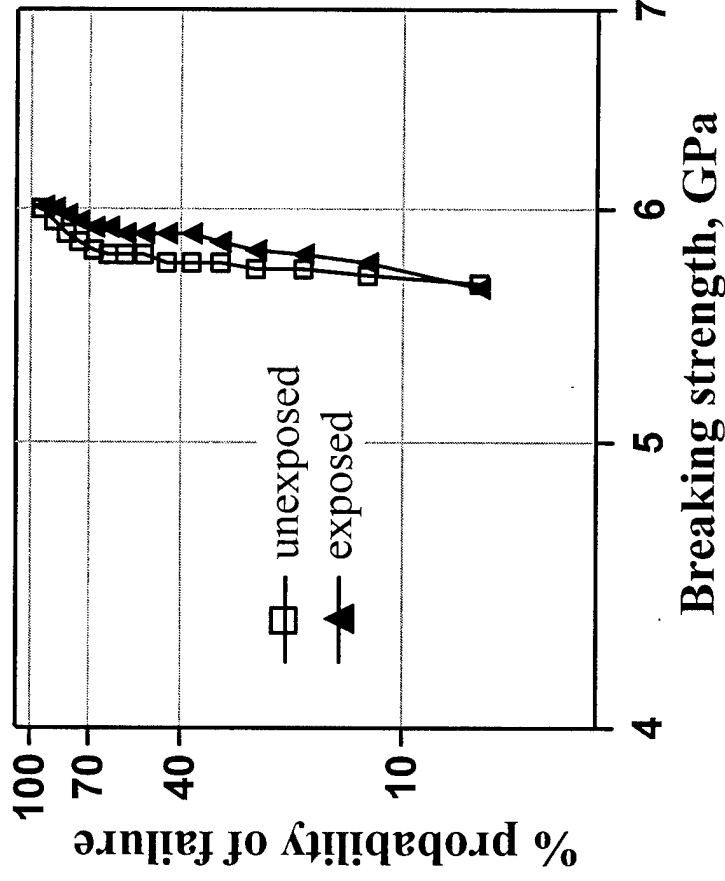
Fiber grating is as strong as ordinary

Chemical and mechanical stripping of fiber jacket



Chemical stripping preserves strength (almost)

10dB Bragg gratings written through the polymer of hydrogen-loaded fiber



Measurements done by S. Semjonov, FORC, Moscow

Starodubov et al, 1997 OSA Tech. Dig. Series, PD1.

Grating lifetime calculation:

$$Lifetime[sec] = \frac{(\sigma_{fail}(1))^{n+1}}{(\sigma_{service})^n} F^{\frac{n+1}{m}}$$

$\sigma_{fail}(1)$ – approximated stress value where all the fiber samples break (probability=1),

$\sigma_{service}$ – service stress value,

F - probability of failure,

n - corrosion susceptibility factor,

m - Weibull shape constant taken from inclination of Weibull plot

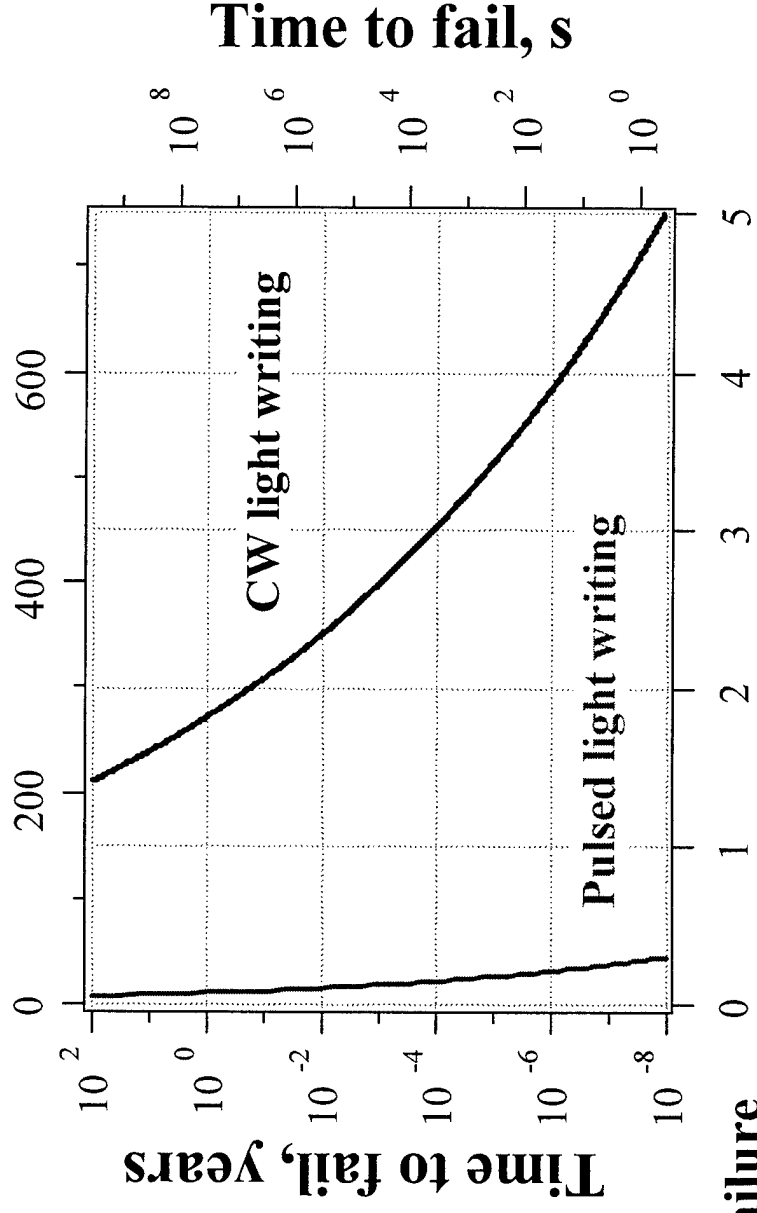
assuming dependence $F = 1 - \exp \left[- \left(\frac{\sigma}{\sigma_0} \right)^m \right]$

*CW written grating: m=57 , n=18 , $\sigma_{fail}(1)$ =3.5 GPa

*Excimer written grating: m=3.8 , n= 13.5, $\sigma_{fail}(1)$ =5.1 GPa

Grating lifetime prediction

Service stress, kpsi



Failure probability

$F=0.0001$

Service stress, GPa

Fiber gratings made
with pulsed light
from excimer lasers
are *not mechanically*
reliable

Data from: T.Volotinen, W. Griffioen, M. Gadonna, H.G.Limberger, "Reliability of Optical Fiber Components", Springer-Verlag London Ltd., 1999.

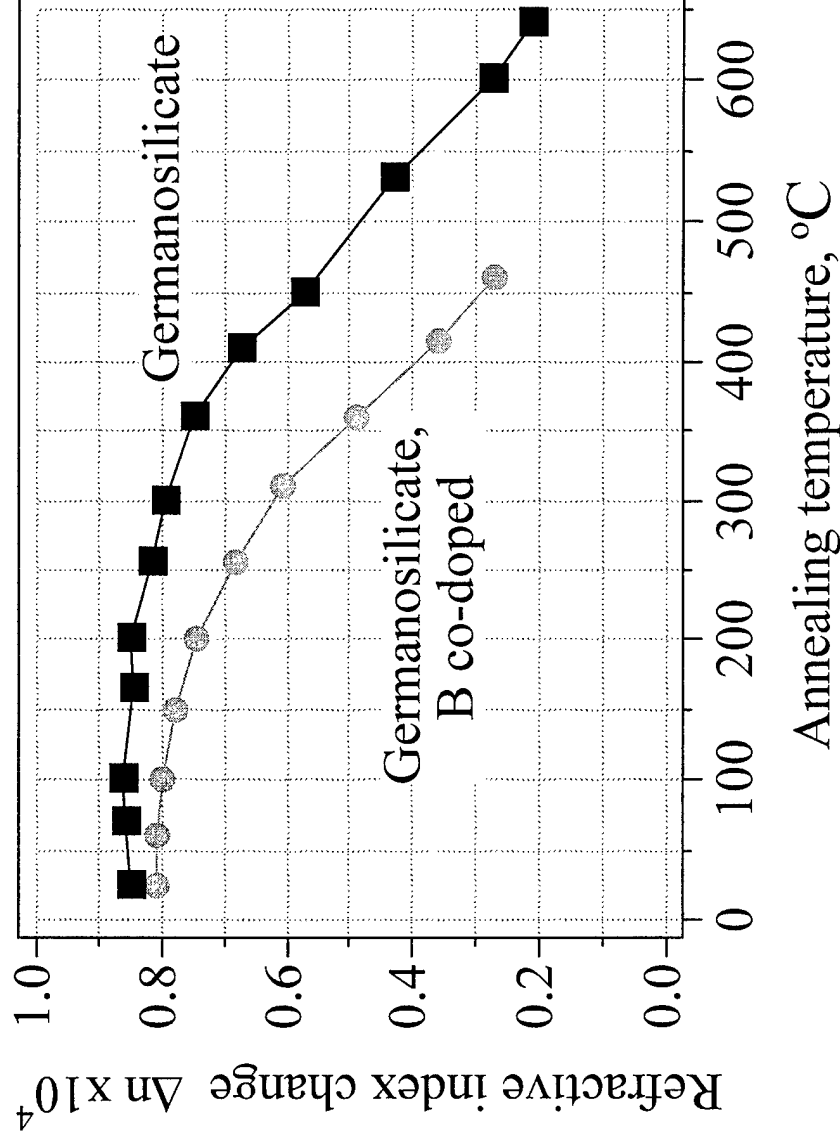
Stability of optical properties

**Fiber gratings decay with time,
optical properties of the fiber gratings change**

Decay increases with temperature

Stability of gratings is crucial for WDM applications

Annealing of gratings with no hydrogen



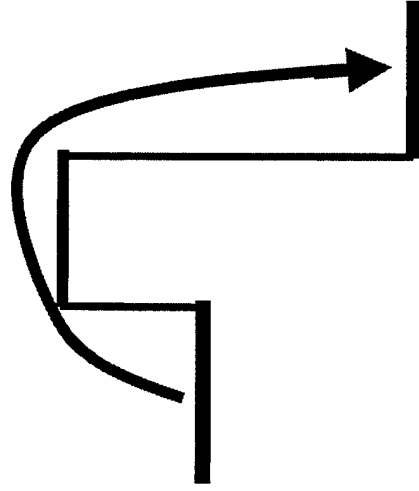
Fiber gratings in B co-doped fibers degrade at lower temperature

Typical grating fabrication procedure through the coating

- 1) ~~Strip the fiber coating~~ —
↓
- 2) Write a grating
↓
- 3) Thermally anneal the grating
↓
- 4) ~~Recoat the fiber~~

How to anneal grating with coating on ?

“Light annealing”: physics

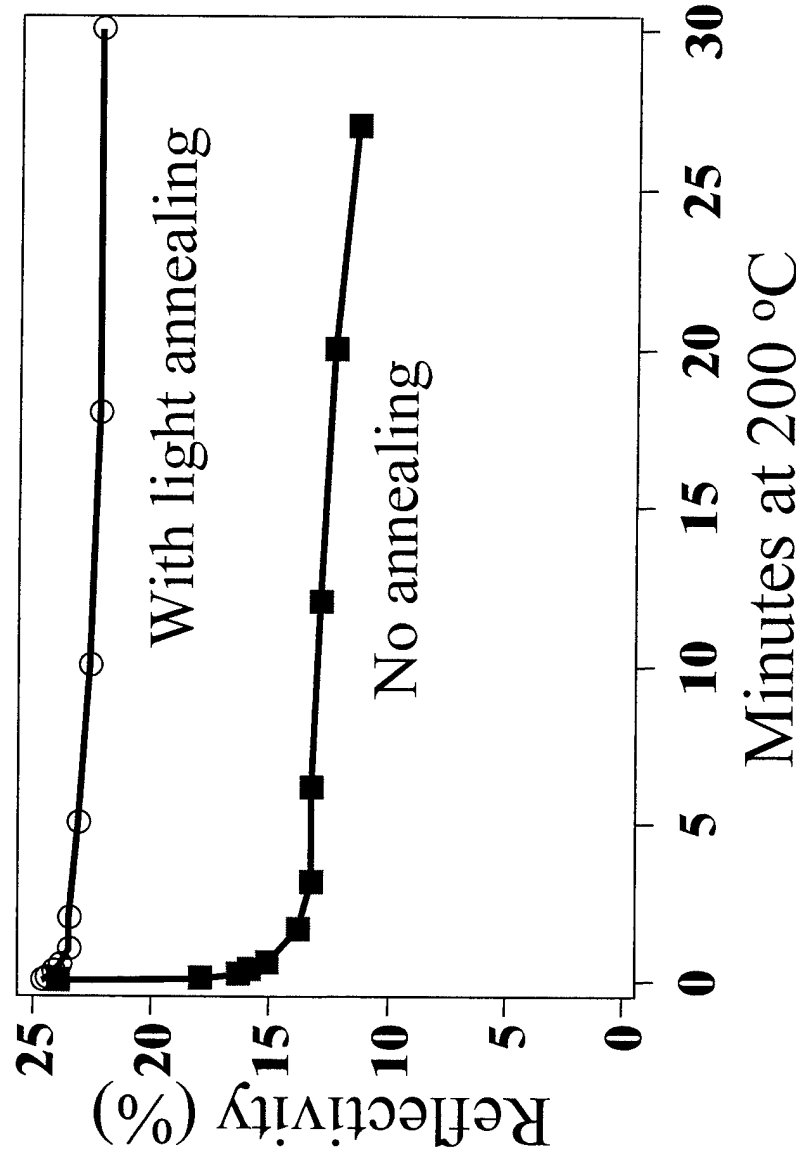


Final state

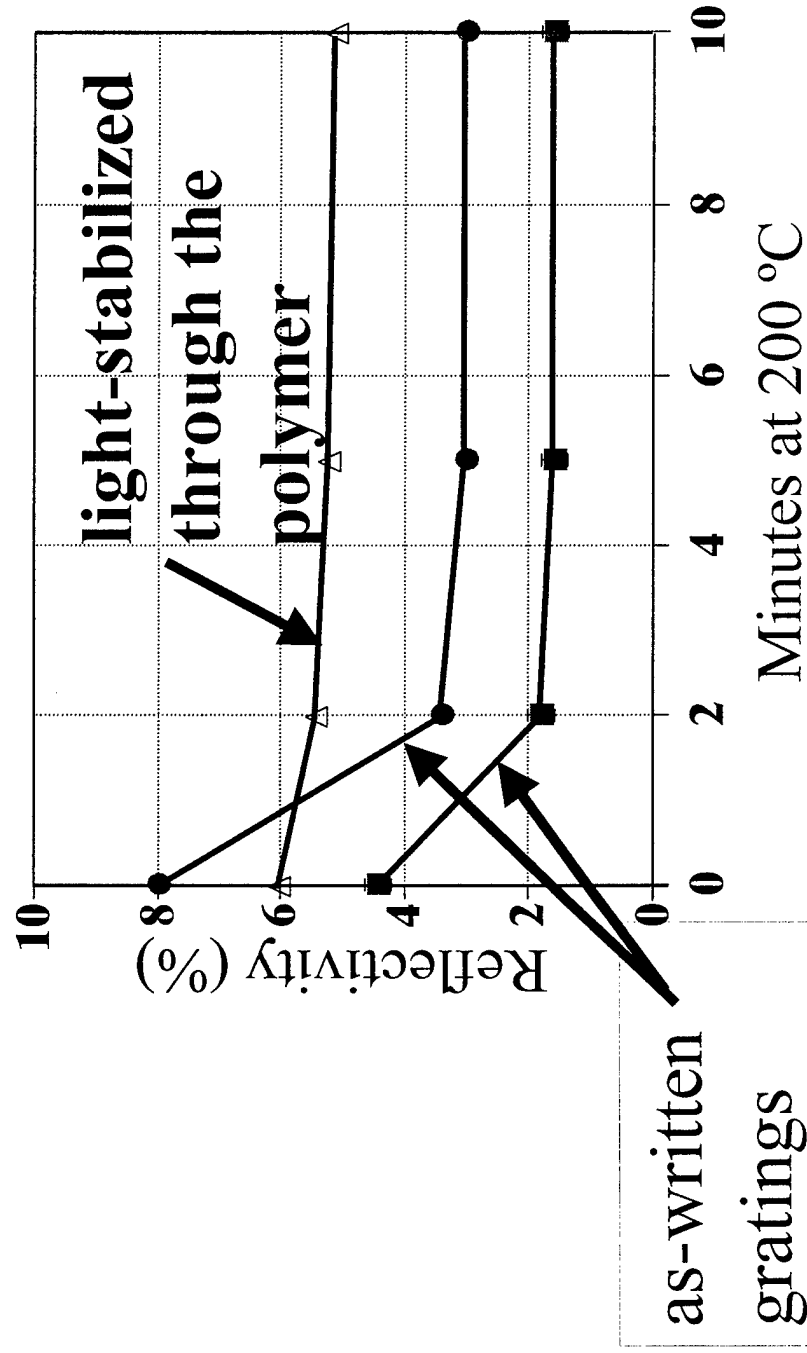
**Defect sites with small
barriers fill first,**

**but are the least
stable thermally.**

“Light annealing” stabilizes reflectivity

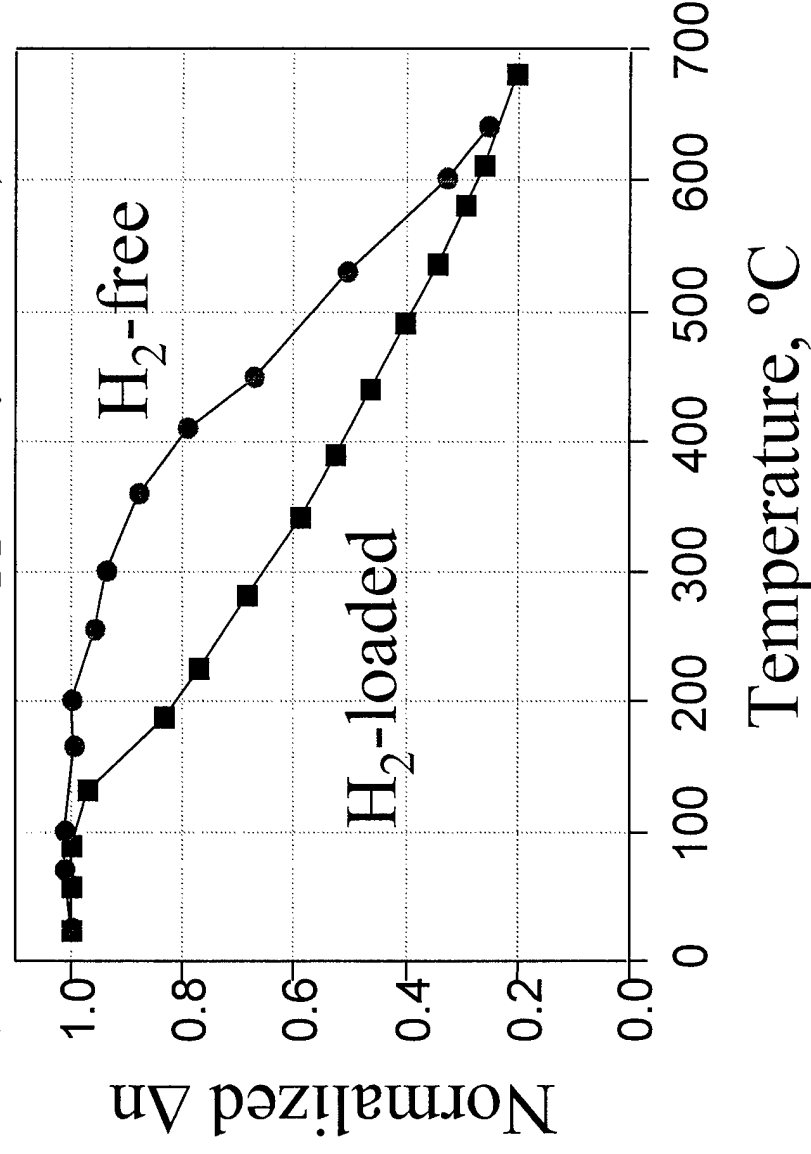


“Light annealing” through polymer

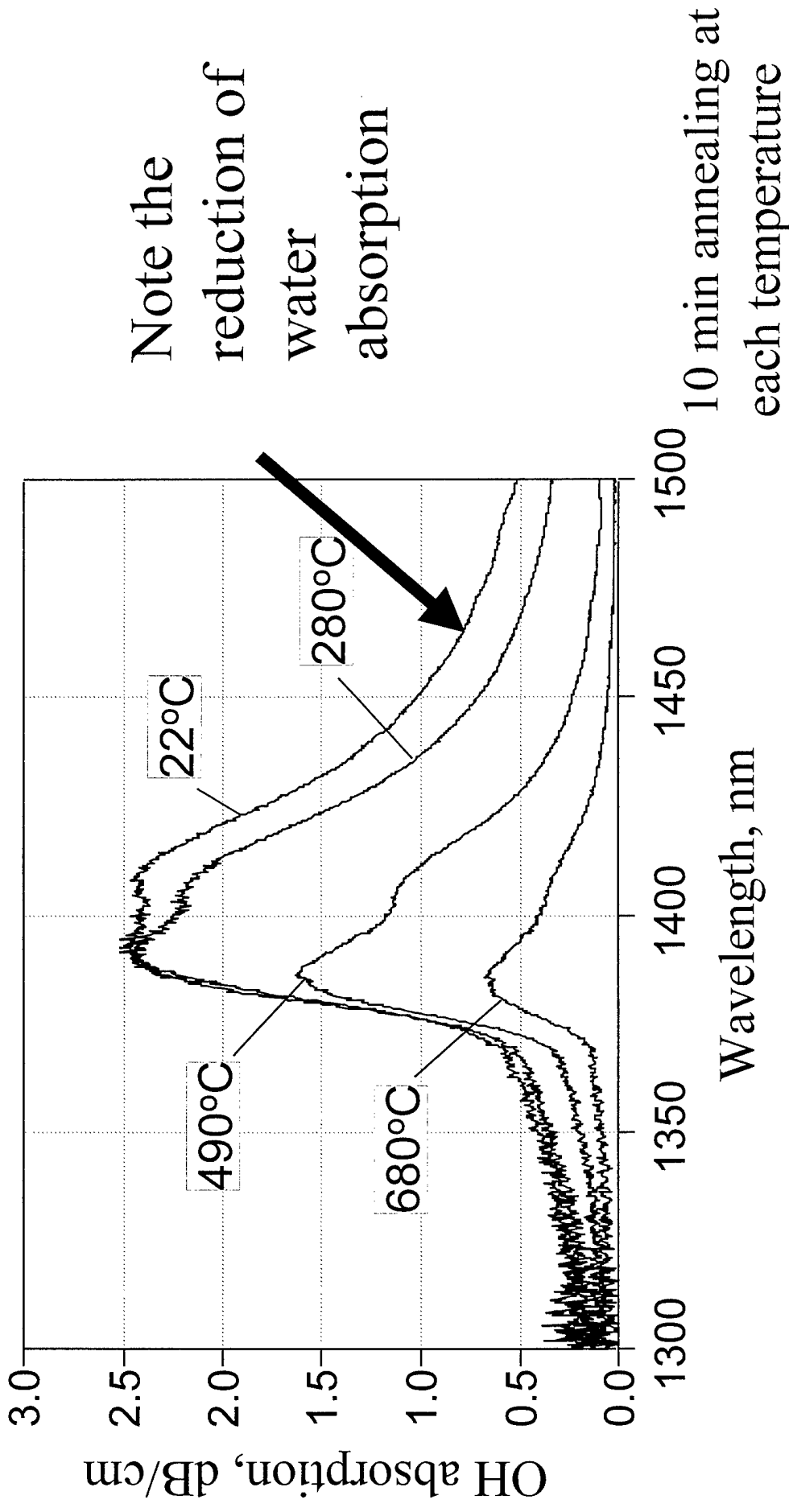


Gratings in H₂-loaded fibers have poor stability at low temperature compared to H₂-free fibers

(Patrick et al., *J. Appl. Phys.*, 1995)

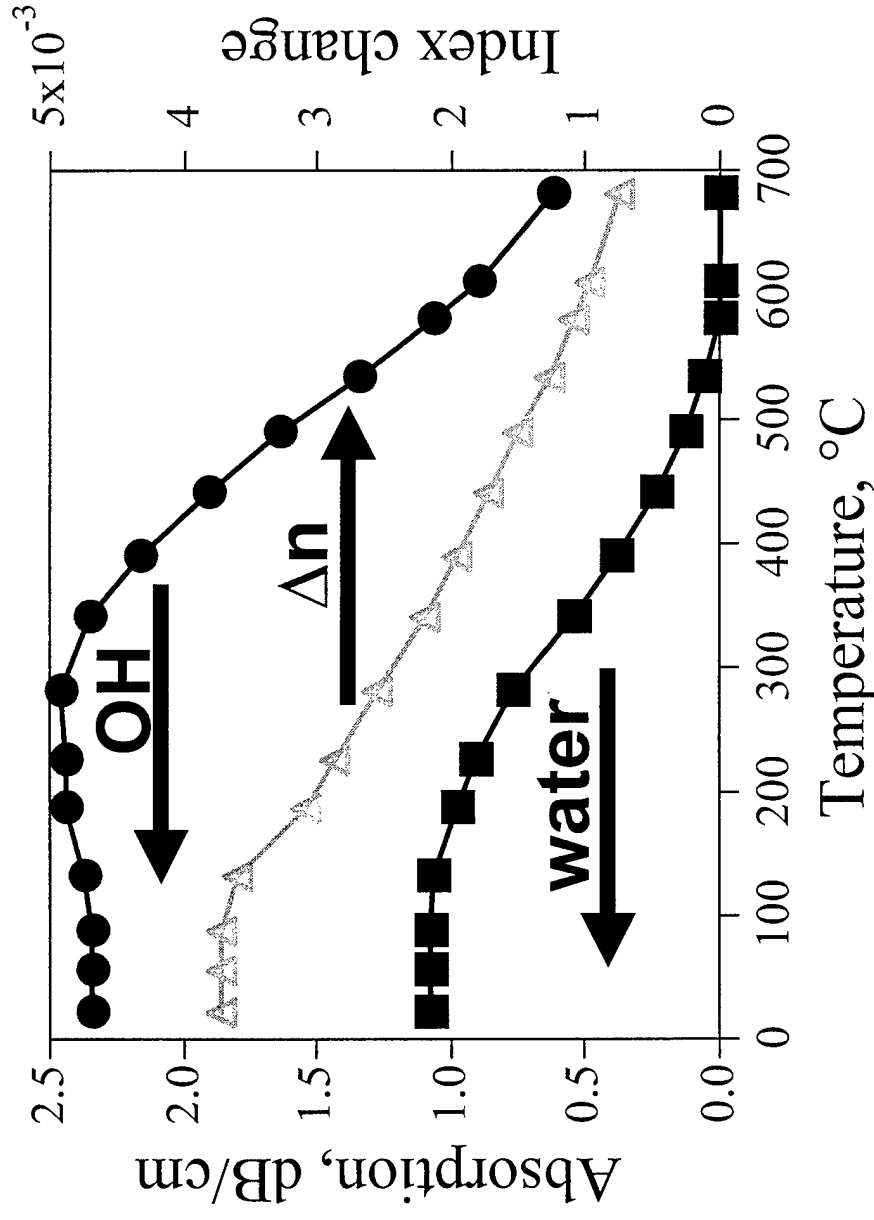


Annealing first removes water, then OH



Grubsky et al, OFC 1999, paper ThD2

Only 30% of index change is left when water is gone



Two important facts to know:

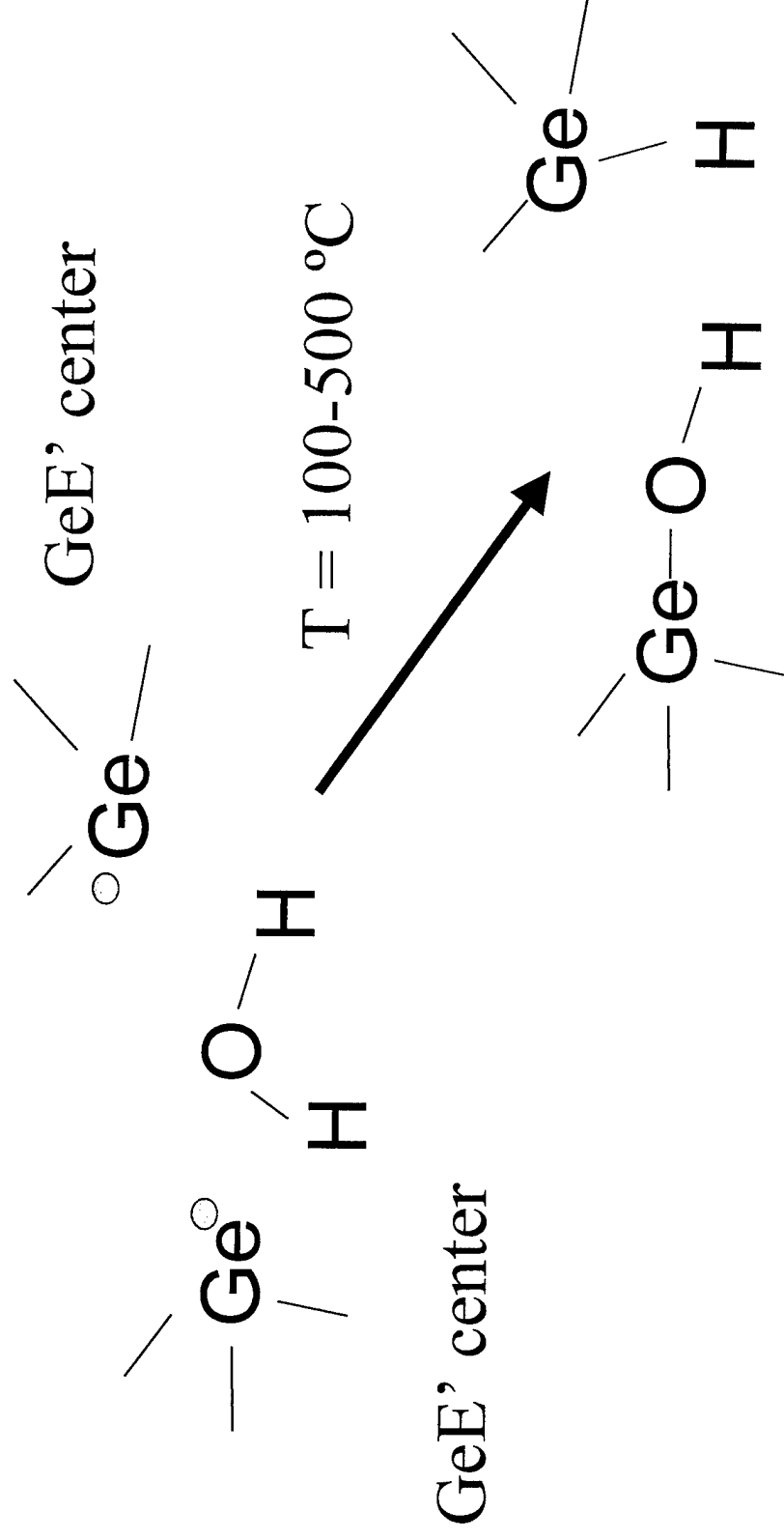
1. At low temperatures ($T < 300\text{ }^{\circ}\text{C}$) water anneals but OH peak remains unchanged.
 2. At high temperatures ($T > 500\text{ }^{\circ}\text{C}$) only OH is present in the core; water is gone.
-

Additional complications

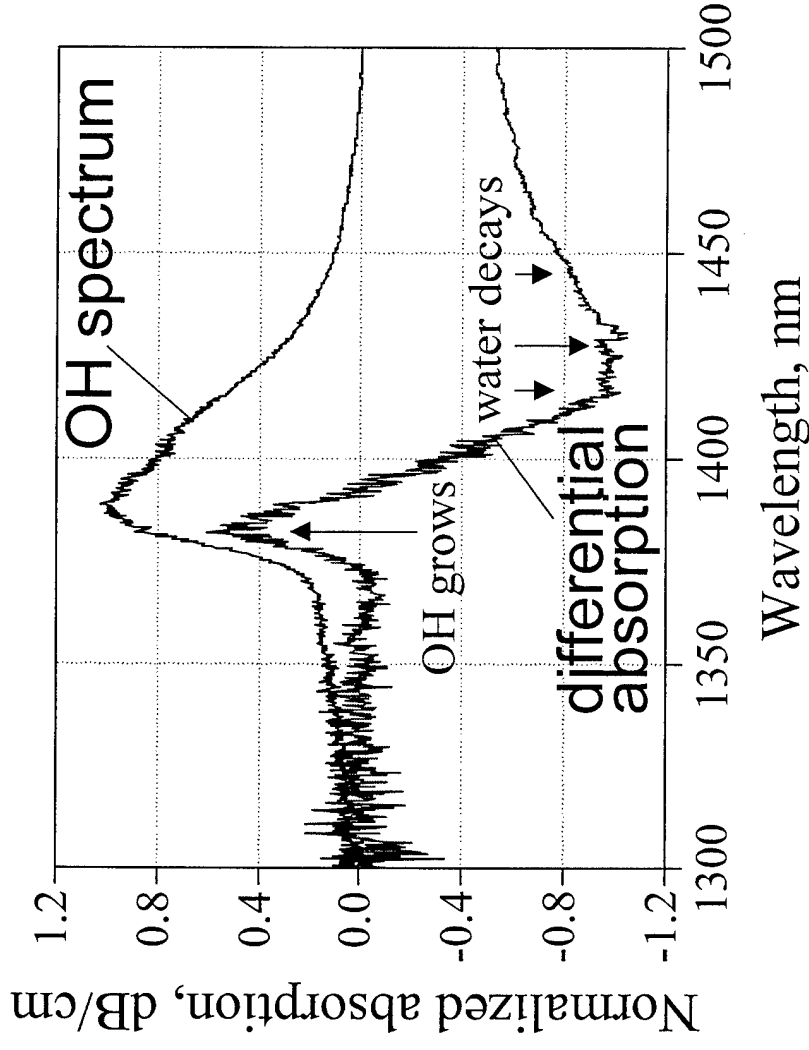
Water cannot leave the fiber core at low temperatures ($T < 300\text{ }^{\circ}\text{C}$) because of its slow diffusion in glass.

Therefore, water and Δn decay at ($T < 300\text{ }^{\circ}\text{C}$) are caused by a chemical reaction.

Decay of Δn may be due to the reaction of water with GeE' centers



Water decay correlates with OH growth at low temperatures (25°C-280 °C)



Grubsky et al, OFC 1999, paper ThD2

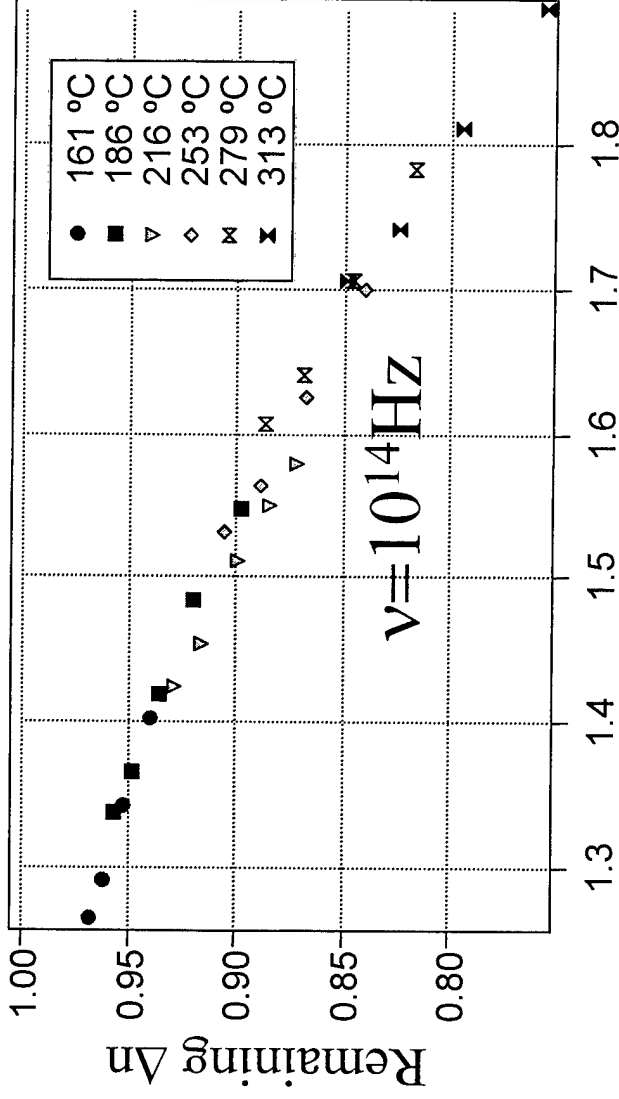
Annealing of hydrogen loaded fibers: summary

1. Photolytical molecular water is the cause of high losses at 1.55 μm and poor thermal stability of gratings written in H_2 -loaded fibers.
 2. Harmful effects can be removed by annealing gratings at temperatures as high as 500 $^{\circ}\text{C}$.
 3. Gratings produced by H_2 -loading are strong but unstable unless annealed at high temperature.
Only *$\sim 1/3$ of their Δn* remains after complete stabilization.
-

Lifetime prediction idea

1. Anneal gratings at several temperatures.
2. Get annealing dependence (energy distribution).
3. Estimate low temperature behavior.

Lifetime prediction curve



Annealing data at different temperatures assembled together to figure out parameter ν

Demarcation energy E_d , eV

$$E_d = k_B T \ln(\nu)$$

$k_B - 1.38e-23 \text{ J K}^{-1}$

Operation temperature

Time to last

Remaining Δn comes from the curve after calculating E_d for given time and temperature

Conclusion

Make sure that the actual grating you get is
reliable enough for your application
