CHAPTER 4

Interfaces

Interface

- An interface between any two phases: a surface across which a discontinuity in one or more of material parameters or properties occurs.
  - For example in lattice parameter, density, elastic modulus, thermal expansion coefficient, strength, fracture toughness, etc.
- The behavior of a composite material is a result of the combined behavior of the:
  - Reinforcement
  - Matrix
  - Reinforcement/matrix interface

Interfacial area

✓**Example:** Calculate the total reinforcement/matrix interface area (per Cm^3) of a 50% fiber reinforced composite (d=10 µm).

- For a cylindrical fiber in a matrix:
  - Surface area per unit volume of a fiber (ignoring the ends)

$$\frac{S}{V} = \frac{2}{R} = \frac{4}{d} \qquad \text{(d = diameter)}$$

  - Fine fibers (diameter ≈ a few µm) can lead to very large interfacial areas
- For a laminated composite made by laminating sheets of two materials of thickness t:

  →The interfacial area α 1/t

- It easily can go as high as 3,000 cm^2/cm^3 in composites

- The reinforcements should not be weakened by flaws because of an adverse interfacial reaction
- The applied load should be effectively transferred from the matrix to the reinforcements via the interface
  - The nature of the interface region under a given set of conditions
  - Wettability of the reinforcement by the matrix
  - The type of bonding between the two components
  - How the characteristics of the interface are affected by temperature, diffusion, residual stresses, and so on.

Wettability

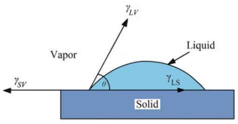
Definition: Ability of a liquid to spread on a solid surface

- The sessile drop experiments: A liquid drop will spread and wet a surface only if this results in a net reduction of free energy of the system.
- Equilibrium of three forces (three specific surface tensions or energies):

  γ_{sv} of the solid/vapor interface

  γ_{ls} of the liquid/solid interface

  γ_{lv} of the liquid/vapor interface



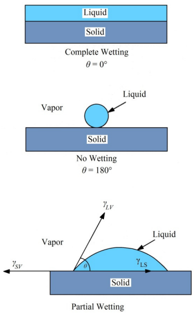
Contact angle, θ

- Young's equation: Equilibrium of forces in horizontal direction gives

$$\gamma_{sl} + \gamma_{lv} \cos \theta = \gamma_{vs}$$

or, $$\cos \theta = (\gamma_{vs} - \gamma_{sl})/\gamma_{lv}$$

  - $\theta = 0°$, perfect wetting
  - $\theta = 180°$, no wetting
  - For $0° < \theta < 180°$, partial wetting



Contact angle

- **Wettability ≠ Bonding**

- A low contact angle, i.e. good wettability, is a necessary but not sufficient condition for strong bonding.

- Contact angle (θ) is a also a function of:
  - Interfacial reactions and contamination
  - Time and temperature of contact
  - Substrate roughness and geometry
  - Humidity
  - Environment
  - …

- **Effect of Surface Roughness**

- The interface between the reinforcement and matrix is never perfectly planar

- Most reinforcements show some degree of roughness

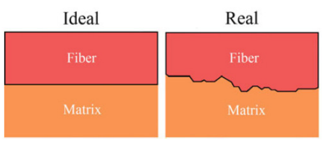


Fig. 4.2 (**a**) An ideal planar interface between reinforcement and matrix. (**b**) A more likely jagged interface between fiber and matrix

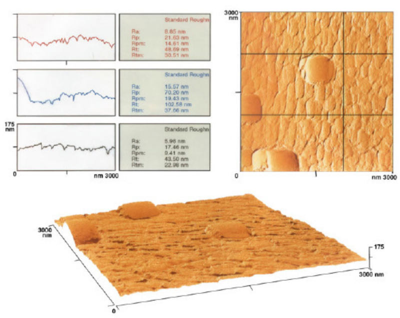


Fig. 4.3 Characterization of surface roughness of a polycrystalline alumina fiber (Nextel 610) by atomic force microscopy. The three profiles on the *left-hand side* correspond to the two horizontal and one vertical lines on the *right-hand side* figure. The *bottom figure* shows a three-dimensional perspective view of surface

- A good wetting is required for the liquid to penetrate the crevices.
- The effect of surface roughness on wettability can be described by *Wenzel's equation*:

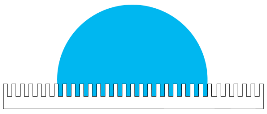
$$\cos\ \theta_0 = r\frac{\gamma_{SV} - \gamma_{LS}}{\gamma_{LV}}$$

- $r = A_{real} / A_{proj}$
  - A_{real}= the real area of the interface
  - A_{proj}= the projected area of the interface.

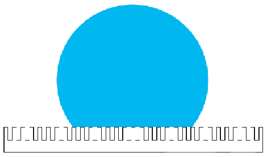
$$Cos\ \theta_{Wenzel} = r\ Cos\ \theta_{Young}$$

- If $0 \le \theta_Y < 90 \rightarrow$ wettability is enhanced by roughness.
- If $90 < \theta_Y \le 180 \rightarrow$ wettability is reduced by roughness.
- If wetting is poor (θ > 90), surface roughness can reduce bonded area and lead to void formation.

- Wenzel model:

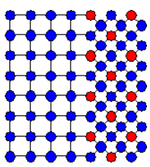


- Cassi-Baxter model:



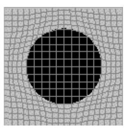
Next assignment!

Crystallographic Nature of Interface



✓ **Coherent interface**:

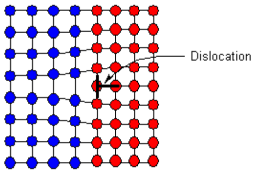
- One-to-one correspondence between lattice planes on the two sides of the interface
- Involves an elastic deformation of the crystals



- Has a lower energy than an incoherent one
  - Example: The interface between GP zones and the Al matrix in Al-Cu system

✓ **Semicoherent interface:**

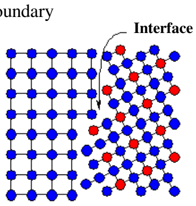
- Only a partial atomic registry
- Small lattice mismatch between the two phases accommodated by the introduction of dislocations at the interface.



- Examples: Interfaces between a precipitate and a matrix , interfaces in some eutectic composites such as NiAl–Cr system

✓ **Incoherent interface:**

- Severe atomic disorder, no matching of lattice planes across the boundary, no continuity of lattice planes across the interface
- No coherency strains, but high boundary energy because of severe atomic disorder at the grain boundary

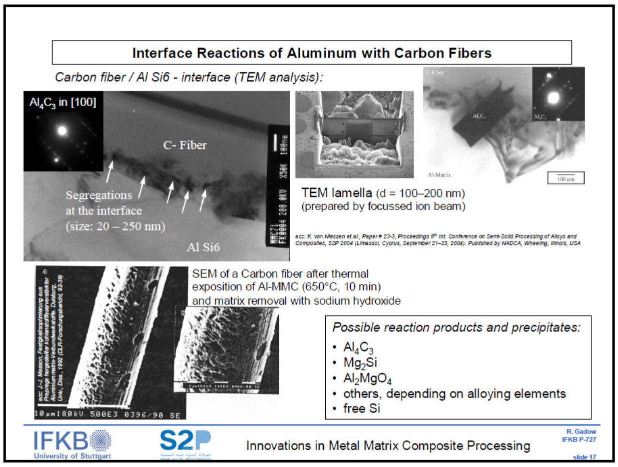


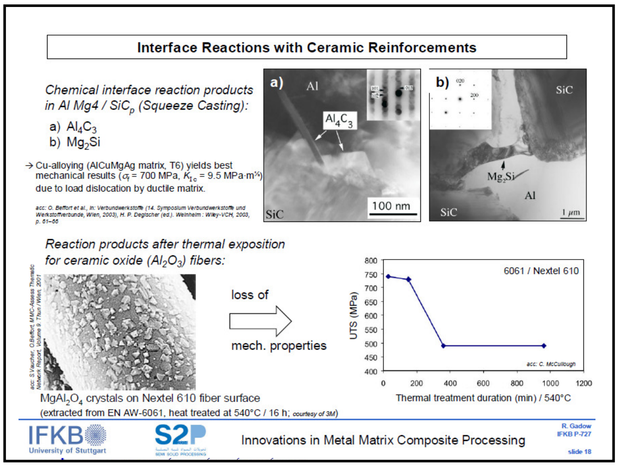
- ***Most of the interfaces*** *that one encounters in fiber, whisker, or particle reinforced composites are* ***incoherent****.*

Interactions at the Interface

- Components of composites are rarely in thermodynamic equilibrium.
  - → A driving force for some **interfacial reaction(s)** leading to a state of thermodynamic equilibrium
- An initially ***planar interface*** can become an ***interfacial zone*** with multiple interfaces resulting from the formation of different intermetallic compounds, interdiffusion, and so on.
- Thermodynamic information can help **predict** the final equilibrium state of the composite:
  - Phase diagrams, reaction kinetics, diffusivities of one constituent in another, ... can provide information about the rate at which the system would tend to attain the equilibrium state.

- In the absence of thermodynamic and kinetic data, **experimental studies** have to be done to determine the compatibility of the components.
- Characteristics of the interfacial zone (different phases in the interfacial zone):
  - Compositional parameter
  - Geometry and dimensions
  - Microstructure and morphology
  - Mechanical, physical, chemical, and thermal characteristics
  - …





- **Fabrication process** can alter the interface characteristics.
- For example, the difference in the CTEs of the two components results in thermal stresses at the interface

→ In MMCs

- The softer component (generally the matrix) will deform plastically.
- High dislocation density observed in the matrix near the interface
- Examples: Cu/W_{fiber} and Al/SiC $_{whisker}$ cast composites and many other MMCs

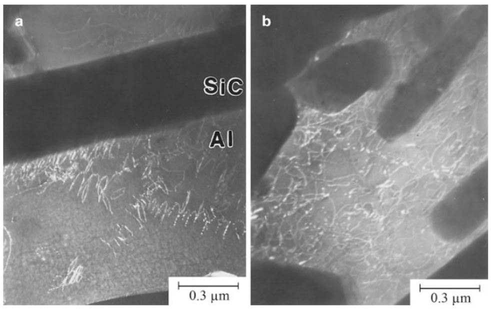


Fig. 6.26 Dislocation distribution in the aluminum matrix of a SiC_w/Al composite: (**a**) inhomogeneous dislocation distribution before testing, (**b**) uniform dislocation distribution after fatigue testing. [From Williams and Fine (1985a), used with permission]

→ In PMCs and CMCs

- The matrix is unlikely to deform plastically in response to the thermal stresses → microcracking

- In powder processing techniques
  - The nature of the powder surface (e.g. an oxide film always present on the surface of powder particles) influences the interfacial interactions and chemical nature of the powder.
- **Topographic characteristics** of the components
  - Affect the degree of atomic contact between the components
  - Geometrical irregularities at the interface (roughness, voids, …) → Stress concentrations

Types of bonding at the interface

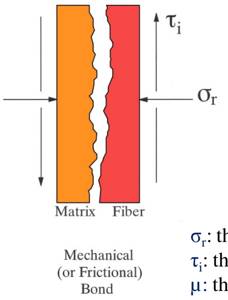
✓ Important types of interfacial bonding:

- Mechanical bonding
- Physical bonding
- Chemical bonding

Types of bonding at the interface

- **Mechanical Bonding**
  - Mechanical keying or interlocking can lead to bonding.
  - Matrix in a composite radially shrinks more than the reinforcement on cooling from a high temperature.
    →gripping of the reinforcement by the matrix even in the absence of any chemical bonding.
  - The matrix penetrating the crevices on the reinforcement surface, by liquid or viscous flow or high temperature diffusion, can also lead to some mechanical bonding.

Mechanical bonding



$$\tau_i = \mu \, \sigma_r$$

σ_r: the radial gripping stress
τ_i: the interfacial shear stress
μ: the coefficient of friction (0.1-0.6)

Mechanical bonding

- In general, mechanical bonding is a low energy bond compared to chemical bonding
  - i.e., the strength of a mechanical bond is lower than that of a chemical bond.
- Pure mechanical bonding alone is not enough in most cases.
  - It could add, in the presence of reaction bonding, to the overall bonding
- Mechanical bonding is efficient in load transfer when the applied force is parallel to the interface
  - The matrix must fill the hills and valleys on the surface of the reinforcement

Mechanical keying: Importance of wettability

- Mechanical keying of the matrix depends on the roughness of the reinforcement.

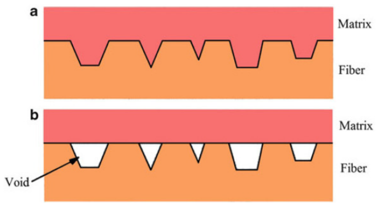


Fig. 4.5 (a) Good mechanical bond. (b) Lack of wettability can make a liquid polymer or metal unable to penetrate the asperities on the fiber surface, leading to interfacial voids

Mechanical keying

- Surface roughness can contribute to bonding only if the liquid matrix wets the reinforcement surface.
- If the matrix (liquid polymer or metal) is unable to penetrate the asperities on the fiber surface, then the matrix will leave interfacial voids on solidification !
- An example of excellent wetting is between WC and cobalt liquid ($\theta = 0$)

Examples of mechanical bonding

- Carbon fiber/epoxy
  - Nitric acid oxidation of carbon fibers
    - Increases specific surface area + good wetting
    - Improved interlaminar shear strength (ILSS) of the composite
- Al_2O_3 / Al composites:
  - There is only mechanical bond between Al_2O_3 and Al.
  - Rough interface: More efficient load transfer from the aluminum matrix to the alumina
- CMCs:
  - Mechanical bonding is preferred over chemical bonding!
  - Roughness-induced gripping at the interface is quite important.

Physical bonding

- Any bonding involving
  - Van der Waals forces,
  - Dipolar interactions, or
  - Hydrogen bonding
- Bond energy is very low, E ~8–16 kJ/mol.

Chemical bonding

- Covalent,
- Ionic, or
- Metallic bonding

- Bond energy is high, E ~ 40 - 400 kJ/mol

Chemical bonding

- Chemical bonding involves:
  - Atomic or molecular transport by diffusional processes,
  - Solid solution and compound formation at the interface,
  - Formation of a interfacial reaction zone having a certain thickness,
  - High energy covalent, ionic, and metallic bonds
- Two main types of chemical bonding:
  - Dissolution bonding
  - Reaction bonding

Chemical Bonding: *Dissolution Bonding*

- Atomic species dissolve into one another at the interface.
- Short-range interaction (at an electronic scale)
- An excellent wettability is of great importance.
- In the absence of an intimate contact, the characteristic short-range interaction cannot occur.
- Surfaces should be treated to remove any impurities, contamination or entrapped air or gas bubbles at the interface

Composite Materials, 2016, BN, IUT, Iran

Chemical bonding: *Reaction bonding*

- Solid solutions and compound formation in the interface zone.
- Atomic, ionic or molecular transport by diffusional processes from one or both of the components to the reaction site (interface)
- Wettability is again of great importance

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Interfacial reactions in MMC

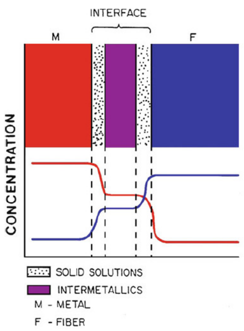

Fig. 4.6 Interface zone in a metal matrix composite showing solid solution and intermetallic compound formation

The reaction products and the reaction rates can vary depending on the matrix composition, reaction time, and temperature.

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Solid solution and compound formation at the interface

• Diffusion theory: For a diffusion-controlled growth

$$x^2 \sim Dt$$
$$D = A \exp(-Q/kT)$$

x = reaction zone thickness,
D = diffusion coefficient,
t = time,
A = pre-exponential constant,
Q = activation energy,
k = Boltzmann's constant
T= temperature in K

$t\uparrow \rightarrow X\uparrow$
$T\uparrow \rightarrow X\uparrow$

Solid solution and compound formation at the interface

- Processing of (cast) MMCs: Both **T** and **t** are generally large
  - → Significant chemical reaction which may adversely affect the behavior of the composite
- Examples: **Al-SiC**

  $4\ Al + 3\ SiC \leftrightarrow Al_4C_3 + 3\ Si$
- → Formation of aluminum carbide (brittle)
- Addition of Si to the matrix (change in melting point, solidification mode, properties, …)
- Control of time, temperature, and initial composition
- High Si content (>10%) alloys are invulnerable

Solid solution and compound formation at the interface

Al-Mg/Al_2O_3

- High levels of Mg in the matrix: MgO formation at the interface

  $3\ Mg + Al_2O_3 \leftrightarrow 3\ MgO + 2\ Al$
- Low levels of Mg in the matrix: Spinel formation at the interface

  $3\ Mg + 4\ Al_2O_3 \leftrightarrow 3\ MgAl_2O_4 + 2\ Al$
- *Controlled amount of reaction* at the interface → Strong interfacial bonding
- Too thick an interaction zone → Adverse effects on properties

Solid solution and compound formation at the interface

Mg-Al/SiC

$$4\ Al\ (\text{in Mg alloy}) + 3\ SiC \leftrightarrow Al_4C_3 + 3\ Si$$

- Formation of aluminum carbide and injection of Si to the matrix → Formation of Mg_2Si in the matrix

Ti-6Al-4V/SiC

TiC and Ti_xSi_y formation on the interface

Optimum Interfacial Bond Strength

- Maximizing the bond strength is not always the goal!
- In CMCs, too strong a bond would cause embrittlement.
- If the interface is as strong or stronger than the reinforcement, it will have the lowest strain-to-failure of the three components (i.e. reinforcement, matrix, and interface)
- The composite will fail when any cracking occurs at a weak spot along the brittle interface → very low toughness

Optimum Interfacial Bond Strength

- Optimum interfacial bond strength: Enhanced toughness, but without a severe penalty on the strength
- Such a composite will have multiple failure sites spread over the interfacial area, which will result in a diffused or global spread of damage, rather than a very local damage.

Tests for Measuring Interfacial Strength

Numerous tests have been devised including:

- Flexural Tests
  - Three and Four-Points Bending
  - Short-Beam Shear Test (Interlaminar Shear Stress Test)
  - Iosipescu Shear Test
- Single Fiber Pullout Tests
- Curved Neck Specimen Test
- Instrumented Indentation Tests
- Fragmentation Test
- Laser Spallation Technique

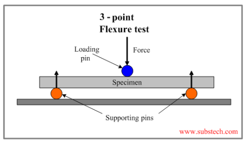
Tests for Measuring Interfacial Strength

Flexural Tests: Very easy to do

- **Three-Point Bending Test:**
  - Bent until the interface fails
  - Maximum tensile stress occurs at the outermost surface.

$$\sigma = \frac{3PS}{2bh^2} \quad \textbf{(Eq. 1)}$$

P = load, S = span,
b = specimen width, h = specimen height



- Fibers parallel to the specimen length

→ measures longitudinal strength of fiber/matrix interface

- Fibers perpendicular to the specimen length

→ measures transverse strength of fiber/matrix interface.

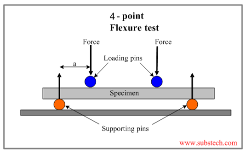
– Maximum shear stress, τ_{max}, occurs at the midplane and is given by

$$\tau_{max} = \frac{3P_{max}}{4bh} \quad \textbf{(Eq. 2)}$$

- **Four-Point Bending Test:**

$$\sigma = \frac{3PS}{4bh^2}$$

S = the outer span



- No transverse shear stresses occur on the cross sections of the beam between the two inner loading points

Short beam shear test
(Interlaminar Shear Stress test - ILSS)

- A special longitudinal three-point bend test with fibers parallel to the length of a very *small bend samples* (S = 5h), ASTM (D2344), can also be used for *laminar composites*
- Dividing max. shear stress (Eq. 2) by max. tensile stress (Eq. 1), we get

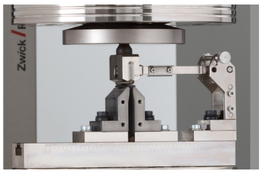
$$\frac{\tau}{\sigma} = \frac{h}{2S}$$

- Important message:
  - Make load span, **S**, very small → Maximize shear stress, **τ**

  → Short specimens are more likely to fail in shear

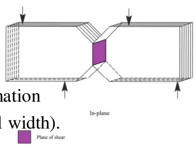
  - Specimen fails under shear with a crack running along the mid-plane.

Short beam shear test
(Interlaminar Shear Stress test - ILSS)

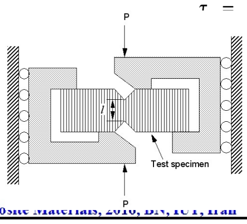


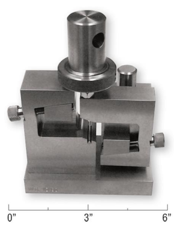
- If the fibers fail in tension before shear-induced failure occurs or if shear and tensile failure occur simultaneously ⇒ **Invalid** test!
- Examine the fracture surface after the test to ensure that the crack is along the interface and not through the matrix!
- For advantages/disadvantages refer to the text book.

Iosipescu shear test



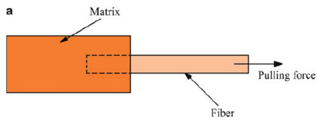
- Double edge-notched sample
- A special type of four-point bend test
- Off-set rollers→ Accentuates shear deformation
- Notch angle=90, Notch depth=22 % of full width).
- Uniform pure shear in the plane
- Ave. shear stress: $\tau = \frac{P}{bh}$





Single fiber pullout test

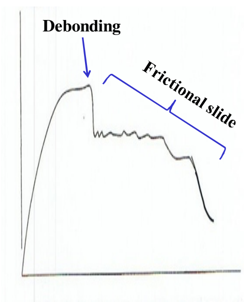
- Can provide useful information about the interface strength in model composite systems.
- Not very useful in case of commercially available composites.



•A portion of fiber, length ℓ, is embedded in a matrix and a pulling tensile force is applied.

•Fabrication of the single fiber pullout test sample is often the most difficult Part.

Single fiber pullout test



- The interfacial shear strength is a function of the coefficient of **friction** and any **normal compressive stress** at the interface.
- The main source of **radial compressive stress** is the **shrinkage of the matrix** during cooling from the processing temperature.
- Effect of different **Poisson's contractions** of fiber and matrix can result in a **radial tensile stress** at the interface (see Chap. 10).

Single fiber pullout test

•The stress required to pull the fiber out of the matrix as a function of the embedded fiber length is measured.

•Must avoid any fiber misalignment and introduction of bending moments.

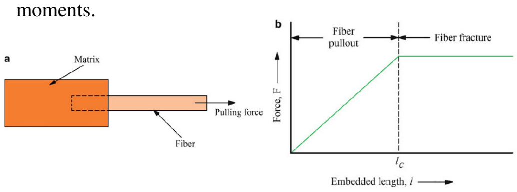


Fig. 4.8 (a) An experimental setup for a single fiber pullout test. A portion of fiber, length l, is embedded in a matrix and a pulling tensile force is applied as shown. (b) The stress required to pull the fiber out of the matrix as a function of the embedded fiber length

Single fiber pullout test

- The stress required to pull the fiber out without breaking it increases linearly with the embedded fiber length, up to a critical length, ℓ_c.
- For embedded fiber lengths ≥ ℓ_c, the fiber will fracture under the action of the tensile stress, **σ**, acting on the fiber.
- The tensile stress, **σ**, acting on the fiber results in a shear stress, **τ**, at the fiber/matrix interface.
- Force balance along the fiber length gives

$$\sigma \pi r^2 = \tau 2 \pi r \ell$$

- For $\ell < \ell_c$, the fiber is pulled out and the interfacial shear strength is given by

$$\tau = \sigma r / 2\ell$$

Instrumented Indentation Tests

- Pointed, conical or rounded indenters can be used to displace a fiber aligned perpendicular to the composite surface.

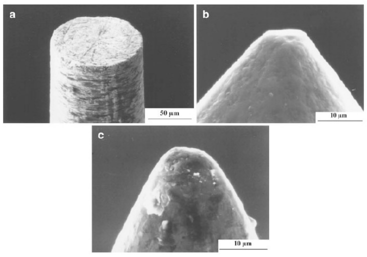


Fig. 4.9 Three different types of indenters: (a) cylindrical with a flat end, (b) conical with a flat end, and (c) pointed (courtesy of J. Janczak-Rusch and L. Rohr)

Instrumented Indentation Tests

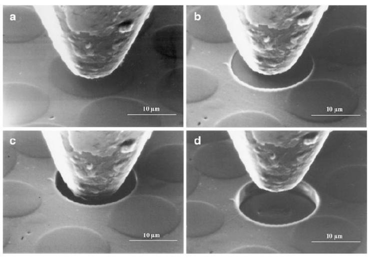


Fig. 4.10 An example of fiber pushin is shown in a series of four photographs. (a) Indenter approaching the fiber (b) touching the fiber (c) fiber pushin (d) lifting of indent (courtesy of J. Janczak-Rusch and L. Rohr)

- By measuring the applied force and displacement, interfacial stress can be obtained.

Instrumented Indentation Tests

- The specimen thickness must be large compared to the fiber diameter, e.g. 1-3 mm.

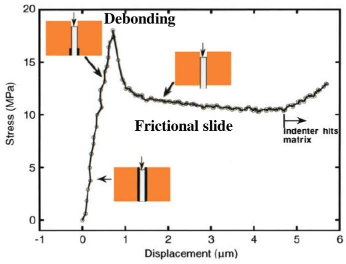


Fig. 4.11 Stress vs. displacement obtained in fiber pushout test (Chawla et al. 2001)

Instrumented Indentation Tests

- **Three-regions** in a valid pushout tests curve:
  - **1st region**: The indenter is in contact with the fiber and the fiber sliding is less than the specimen thickness t.
  - **2nd region**: A horizontal region in which fiber sliding length is greater than or equal to the sample thickness.
  - **3rd region**: The indenter comes in contact with the matrix.
- In the first region, the fiber is elastically compressed by the indenter load over the debonded length, which is assumed to be dependent on the interfacial friction.
- The axial load on the indenter is assumed to be balanced by the frictional stress at the interface, and the effect of radial expansion during indentation is neglected.

Instrumented Indentation Tests

- In the horizontal region, **the interfacial shear stress** is given by:

$$\tau_i = \frac{P}{2\pi r t}$$

P: the applied load
r: the fiber radius
t: the specimen thickness

Other Tests

- Curved Neck Specimen Test
- Fragmentation Test
- Laser Spallation Technique