

Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials VI

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Edited by
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Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials VI

*A Collection of Papers Presented at the
36th International Conference on Advanced
Ceramics and Composites
January 22–27, 2012
Daytona Beach, Florida*



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Preface

The Sixth International Symposium on Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials and Systems (APMT) was held during the 36th International Conference on Advanced Ceramics and Composites, in Daytona Beach, FL, January 22 - 27, 2012. The aim of this international symposium was to discuss global advances in the research and development of advanced processing and manufacturing technologies for a wide variety of non-oxide and oxide based structural ceramics, particulate and fiber reinforced composites, and multifunctional materials. This year's symposium also honored Professor R. Judd Diefendorf, Clemson University, USA, recognizing his outstanding contributions to science and technology of advanced ceramic fibers, carbon-carbon, and ceramic matrix composites and his tireless efforts in promoting their wide scale industrial applications. A total of 68 papers, including invited talks, oral presentations, and posters, were presented from 15 countries (USA, Japan, Germany, China, Korea, France, Australia, Denmark, Estonia, India, Italy, Luxembourg, Serbia, Slovenia, and Taiwan). The speakers represented universities, industry, and research laboratories.

This issue contains 17 invited and contributed papers, all peer reviewed according to The American Ceramic Society review process. The latest developments in processing and manufacturing technologies are covered, including green manufacturing, smart processing, advanced composite manufacturing, rapid processing, joining, machining, and net shape forming technologies. These papers discuss the most important aspects necessary for understanding and further development of processing and manufacturing of ceramic materials and systems.

The editors wish to extend their gratitude and appreciation to all the authors for their cooperation and contributions, to all the participants and session chairs for their time and efforts, and to all the reviewers for their valuable comments and suggestions. Financial support from the Engineering Ceramic Division and The American Ceramic Society is gratefully acknowledged. Thanks are due to the staff of the meetings and publication departments of The American Ceramic Society for their invaluable assistance.

We hope that this issue will serve as a useful reference for the researchers and technologists working in the field of interested in processing and manufacturing of ceramic materials and systems.

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Introduction

This issue of the Ceramic Engineering and Science Proceedings (CESP) is one of nine issues that has been published based on content presented during the 36th International Conference on Advanced Ceramics and Composites (ICACC), held January 22–27, 2012 in Daytona Beach, Florida. ICACC is the most prominent international meeting in the area of advanced structural, functional, and nanoscopic ceramics, composites, and other emerging ceramic materials and technologies. This prestigious conference has been organized by The American Ceramic Society's (ACerS) Engineering Ceramics Division (ECD) since 1977.

The 36th ICACC hosted more than 1,000 attendees from 38 countries and had over 780 presentations. The topics ranged from ceramic nanomaterials to structural reliability of ceramic components which demonstrated the linkage between materials science developments at the atomic level and macro level structural applications. Papers addressed material, model, and component development and investigated the interrelations between the processing, properties, and microstructure of ceramic materials.

The conference was organized into the following symposia and focused sessions:

Symposium 1	Mechanical Behavior and Performance of Ceramics and Composites
Symposium 2	Advanced Ceramic Coatings for Structural, Environmental, and Functional Applications
Symposium 3	9th International Symposium on Solid Oxide Fuel Cells (SOFC): Materials, Science, and Technology
Symposium 4	Armor Ceramics
Symposium 5	Next Generation Bioceramics

Symposium 6	International Symposium on Ceramics for Electric Energy Generation, Storage, and Distribution
Symposium 7	6th International Symposium on Nanostructured Materials and Nanocomposites: Development and Applications
Symposium 8	6th International Symposium on Advanced Processing & Manufacturing Technologies (APMT) for Structural & Multifunctional Materials and Systems
Symposium 9	Porous Ceramics: Novel Developments and Applications
Symposium 10	Thermal Management Materials and Technologies
Symposium 11	Nanomaterials for Sensing Applications: From Fundamentals to Device Integration
Symposium 12	Materials for Extreme Environments: Ultrahigh Temperature Ceramics (UHTCs) and Nanolaminated Ternary Carbides and Nitrides (MAX Phases)
Symposium 13	Advanced Ceramics and Composites for Nuclear Applications
Symposium 14	Advanced Materials and Technologies for Rechargeable Batteries
Focused Session 1	Geopolymers, Inorganic Polymers, Hybrid Organic-Inorganic Polymer Materials
Focused Session 2	Computational Design, Modeling, Simulation and Characterization of Ceramics and Composites
Focused Session 3	Next Generation Technologies for Innovative Surface Coatings
Focused Session 4	Advanced (Ceramic) Materials and Processing for Photonics and Energy
Special Session	European Union – USA Engineering Ceramics Summit
Special Session	Global Young Investigators Forum

The proceedings papers from this conference will appear in nine issues of the 2012 Ceramic Engineering & Science Proceedings (CESP); Volume 33, Issues 2-10, 2012 as listed below.

- Mechanical Properties and Performance of Engineering Ceramics and Composites VII, CESP Volume 33, Issue 2 (includes papers from Symposium 1)
- Advanced Ceramic Coatings and Materials for Extreme Environments II, CESP Volume 33, Issue 3 (includes papers from Symposia 2 and 12 and Focused Session 3)
- Advances in Solid Oxide Fuel Cells VIII, CESP Volume 33, Issue 4 (includes papers from Symposium 3)
- Advances in Ceramic Armor VIII, CESP Volume 33, Issue 5 (includes papers from Symposium 4)

- Advances in Bioceramics and Porous Ceramics V, CESP Volume 33, Issue 6 (includes papers from Symposia 5 and 9)
- Nanostructured Materials and Nanotechnology VI, CESP Volume 33, Issue 7 (includes papers from Symposium 7)
- Advanced Processing and Manufacturing Technologies for Structural and Multifunctional Materials VI, CESP Volume 33, Issue 8 (includes papers from Symposium 8)
- Ceramic Materials for Energy Applications II, CESP Volume 33, Issue 9 (includes papers from Symposia 6, 13, and 14)
- Developments in Strategic Materials and Computational Design III, CESP Volume 33, Issue 10 (includes papers from Symposium 10 and from Focused Sessions 1, 2, and 4)

The organization of the Daytona Beach meeting and the publication of these proceedings were possible thanks to the professional staff of ACerS and the tireless dedication of many ECD members. We would especially like to express our sincere thanks to the symposia organizers, session chairs, presenters and conference attendees, for their efforts and enthusiastic participation in the vibrant and cutting-edge conference.

ACerS and the ECD invite you to attend the 37th International Conference on Advanced Ceramics and Composites (<http://www.ceramics.org/daytona2013>) January 27 to February 1, 2013 in Daytona Beach, Florida.

MICHAEL HALBIG AND SANJAY MATHUR
Volume Editors
July 2012

Contents

Preface	vii
Introduction	ix
Contribution to the Understanding of the Microstructure of First Generation Si-C-O Fibers	1
Francis Teyssandier, Géraldine Puyoo, Stéphane Mazerat, Georges Chollon, René Pailler, and Florence Babonneau	
The Control of Interphases in Carbon and Ceramic Matrix Composites	11
Gérard Vignoles, René Pailler, and Francis Teyssandier	
High Volume Production for High Performance Ceramics	25
William J. Walker, Jr.	
Low Pressure Injection Molding of Advanced Ceramic Components with Complex Shapes for Mass Production	35
Eugene Medvedovski and Michael Peltsman	
Ceramic Injection Molding Using a Partially Water-Soluble Binder System: Effect of Back-Bone Polymers on the Process	53
Oxana Weber and Thomas Hanemann	
Green-Conscious Ceramic Injection Molding	63
Oxana Weber and Thomas Hanemann	
Shaping of Large-Sized Sputtering Targets	73
Alfred Kaiser	

TEM Observation of the Ti Interlayer between SiC Substrates during Diffusion Bonding H. Tsuda, S. Mori, M. C. Halbig, and M. Singh	81
Joining of Alumina by Using of Polymer Blend and Aluminum Ken'ichiro Kita, Naoki Kondo, Hideki Kita, and Yasuhisa Izutsu	91
Diffusion Bonding of Rigid Alumina Pieces using Porous Alumina Interlayers Hiroyuki Miyazaki, Mikinori Hotta and Hideki Kita, and Yasuhisa Izutsu	99
Influence of Joining Pressure and Surface Roughness on Flexural Strength of Joined Boron Carbide Ceramics Kiyoto Sekine, Takeshi Kumazawa, Wu-Bian Tian, Hideki Hyuga, and Hideki Kita	105
Laser Machining of Melt Infiltrated Ceramic Matrix Composite D. C. Jarmon, G. Ojard, and D. Brewer	113
Fabrication of Dendritic Electrodes for Solid Oxide Fuel Cells by using Micro Stereolithography Naoki Komori, Satoko Tasaki, and Soshu Kiriwara	123
Ion-Exchange Properties of Nano Zeolite a Prepared by Bead Milling and Post-Milling Recrystallization Method Toru Wakiwara, Ryuma Ichikawa, Junichi Tatami, Katsutoshi Komeya, Takeshi Meguro	129
The Role of Milling Liquids in Processing of Metal-Ceramic-Precursor Powders Nadja Holstein, Katharina Wiegandt, Florian Holleyn, Jochen Kriegesmann, Michael R. Kunze, Joachim Scholz, and Rolf Janssen	135
Quantitative Validation of a Multi-Scale Model of Pyrocarbon Chemical Vapor Infiltration from Propane G. L. Vignoles, W. Ros, G. Chollon, F. Langlais, and C. Germain	147
Numerical Analysis of Fracture Behavior in Anisotropic Microstructures Hisashi Serizawa, Seigo Tomiyama, Tsuyoshi Hajima, and Hidekazu Murakawa	159
Author Index	169

CONTRIBUTION TO THE UNDERSTANDING OF THE MICROSTRUCTURE OF FIRST GENERATION SI-C-O FIBERS

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ABSTRACT:

As compared to the most recent SiC-based ceramic fibers, first generation fibers include a significant amount of oxygen and free carbon. Though a large number of papers have been devoted to understanding the microstructure of these fibers, their composition and microstructure are still controversial. This communication is intended to propose a microstructure description of these fibers according to their composition in the Si-C-O isothermal section of the phase diagram. The proposed microstructure is deduced from a large set of characterizations including XRD, Raman spectroscopy, RMN, and elemental analysis. Three Nicalon and four Tyranno fibers are thus characterized. Their fracture behavior is also described.

KEYWORDS: Si-C-O fibers, Fiber composition, Fiber microstructure.

INTRODUCTION

Ceramic matrix composites (CMC) were first developed for applications in severe environments: at high temperature, under mechanical stress, in oxidative environments and even under radiations. Such demanding applications require very high properties that are obtained by use of high performance materials. New trends are to develop CMC materials for very long life time applications but at lower temperatures and with a reduced stress level. Such CMC that are aimed at being used in civil aeronautics (aircraft engine plug or exhaust...), do not require the most capable third generation fibers. Instead, the less demanding parts in terms of performance can be designed with less expensive fibers of the first generation. The aim of the present paper is to compare the properties of a variety of first generation silicon carbide fibers.

Since the pioneering work of Yajima et al.¹ in the seventies, the SiC fibers have been improved in order to increase the creep and oxidation resistance². Three families of SiC fibers have been successively developed from the first generation that included carbon and oxygen in excess, to the third generation, which is composed of almost pure silicon carbide. The durability of SiC/SiC composites is at last related to the fibers lifetime, i.e., their sensitivity to oxidation or subcritical crack growth. This latter mechanism is largely influenced by the composition and the structure of the Si-O-C fibers and is expected to be accelerated by the presence of free carbon. The percentage of oxygen and the SiC grain size may also have a significant influence on the fiber reactivity. We therefore undertook the characterization of a wide variety of first generation silicon carbide fibers in order to provide a detailed description of their microstructure and properties.

The microstructure of first generation SiC fibers is globally known. It can be described as a continuum consisting of glassy silicon oxycarbide binding pure β -SiC nanocrystals and including free carbon nanodomains called basic structural units (BSU)³⁻⁸.

Many authors have contributed to the assessment of the microstructure of the various phases composing the Si-O-C fibers. In 1989, Laffon et al.³ studied the NicalonTM NG100 and NG200 fibers mainly by EXAFS and X-ray diffraction and proposed a model of structure including a continuum of β -SiC nanocrystals embedded in a Si-C-O glass consisting of mixed SiO_xC_y ($x+y=4$) tetrahedral environments. Carbon BSU are described as aggregates composed of graphene layers with edges saturated with hydrogen, i.e. without any chemical bonding with the Si-O-C continuum. In 1993, Le

Coustumer et al.⁵ studied the Nicalon fiber NLM202. They determined the following composition: 55wt% of β -SiC crystals, 40wt% of amorphous $\text{SiO}_{1.15}\text{C}_{0.85}$ and 5wt% of free carbon. Knowing the percentage of free carbon and the atomic chemical composition, they calculated the proportion of each phase. An average grain size of 1.6nm was measured using (111)-SiC Dark Field transmission imaging (DF-TEM).

In 1996 Bodet et al.⁹ calculated phase proportions and composition of the fiber Nicalon NLP201 knowing atomic chemical composition and $\text{SiC}/\text{SiO}_x\text{C}_y$ ratio as determined by XPS analysis (from the various Si2p peak components). An average β -SiC grain size of 2.7 nm was measured using (111)-SiC DF-TEM image.

More recently, by use of density functional theory, P. Kroll calculated the structure and properties of amorphous silicon oxycarbide glasses, pure¹⁰ or including a free carbon phase¹¹.

RESULTS AND DISCUSSION

We studied the following first generation SiC fibers: Nicalon NLM202 and 207 from Nippon Carbon, and Tyranno S, ZMI, LoxM from UBE. We also studied the Tyranno AM and Nicalon NLP101 which are not currently commercialized. Their composition and structure are nevertheless interesting to compare to other fibers.

The atomic composition of the whole fiber was first determined by chemical analysis. We then successively studied the β -SiC nanocrystallites by both X-ray diffraction (XRD) and high resolution transmission electron microscopy (HRTEM). The pure Si-C-O glass phase has the composition $\text{SiC}_x\text{O}_{2(1-x)}$. Its composition was both deduced from the atomic composition and by nuclear magnetic resonance (NMR) ^{29}Si analysis. The amount of free carbon was both deduced from the difference between the atomic composition of the fiber and the composition of the Si-C-O continuum, as well as from ^{13}C -NMR analysis. We also characterized the in-plane disorder of carbon BSU by Raman spectroscopy.

Elemental Composition of Fibers

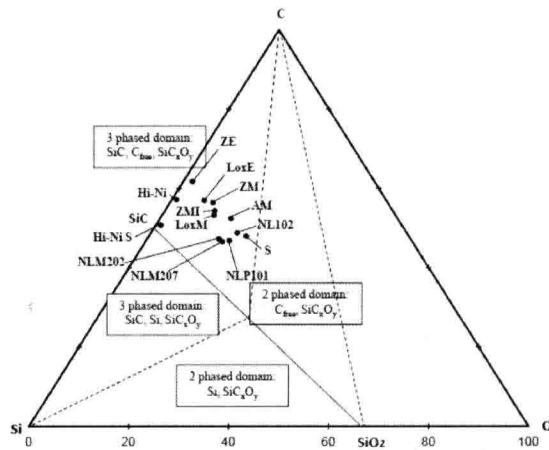


Figure 1: composition of first generation SiC fibers presented in the ternary section of the Si-O-C phase diagram.

The elemental composition of fibers was measured by chemical analysis at the CNRS facility in Solaize. The content of silicon, as well as of Zr, Ti and Al (that are present in limited amounts), was determined by Inductively Coupled Plasma - Atomic Emission Spectroscopy. The carbon concentration was deduced from the amount of CO_2 resulting from oxidation. The oxygen concentration of fibers was deduced from the amount of carbon monoxide formed by reaction at high temperature between ground fibers and the carbon crucible in which they are disposed. Hydrogen was deduced from the amount of water vapor formed by oxidation. The compositions of the fibers are plotted in the isothermal Si-C-O ternary section in figure 1. The Tyranno S fiber has the highest oxygen content and also the largest amount of amorphous $\text{SiC}_x\text{O}_{2(1-x)}$ phase.

Phase Composition of the Fibers: Assumptions

The pure Si-C-O amorphous phase which is composed of mixed tetrahedral environments SiO_xC_y ($X+Y=4$) can be described using a rule of mixture between silica and silicon carbide similar to: $\text{SiC}_x\text{O}_{2(1-x)} = x \text{ SiC} + (1-x) \text{ SiO}_2$. We assumed that the so called "free carbon" phase, embedded into the continuum is not bonded to the amorphous $\text{SiC}_x\text{O}_{2(1-x)}$ glass nor the $\beta\text{-SiC}$ grains⁷. We could thus infer that all the oxygen atoms are bonded to silicon, the remaining silicon being bonded to carbon. Carbon atoms that are not bonded to sp^3 -silicon are hence part of the free carbon domains ($\text{sp}^2\text{-C}$).

Composition of the Si-C-O Continuum

Composition of the Si-C-O continuum ($\beta\text{-SiC} + \text{amorphous silicon oxycarbide}$) was deduced from the atomic composition measured by chemical analysis on each type of fiber. It was calculated according to the formula $\text{SiC}_x\text{O}_{2(1-x)}$ on grounds of assumptions described in the preceding paragraph.

The composition was also measured by nuclear magnetic resonance (NMR) analysis of silicon (^{29}Si) carried out on grounded fibers. NMR analysis enabled us to characterize quantitatively the proportion of the various allowed tetrahedral environments of silicon: SiC_4 , SiC_3O , SiC_2O_2 , SiCO_3 and SiO_4 . The recorded spectra were decomposed into five peaks, one for each silicon environment, the amount of each environment being proportional to the surface of the corresponding peak. The related chemical shifts are observed in the following order: $\delta(\text{SiC}_3\text{O}) > \delta(\text{SiC}_4) > \delta(\text{SiC}_2\text{O}_2) > \delta(\text{SiO}_3\text{C}) > \delta(\text{SiO}_4)$. It is interesting to notice that the chemical shift of the SiC_3O environment is not, as expected, located between those of SiC_4 and SiC_2O_2 , due to a non-linear dependence of the chemical shift (δ) to the charge (q) of atoms.

Compositions obtained by both approaches are in good accordance (figure 2). This result validates our assumptions and especially the absence of bonding between BSU and their neighboring phases.

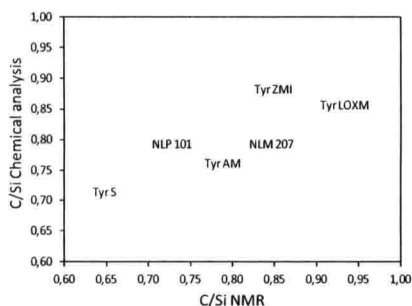


Figure 2: C/Si ratio in the Si-C-O continuum of the various fibers: comparison between calculations based on atomic composition and NMR measurements

NMR measurements revealed that both Nicalon NLP101 and NLM207, as well as Tyranno S and AM include a significant amount of SiO₄ environments (11% for Tyranno S and Nicalon NLP101). This observation provides evidence of a bulk silicon oxycarbide phase between the β-SiC grains and the BSU.

SiC Characterization by X-Ray Diffraction

The amount of crystallized β-SiC phase included in each fiber was determined by X-ray diffraction, carried out on grounded fibers, and including an internal silicon standard. The quantitative analysis was performed by comparing the areas of SiC and Si peaks after calibration with a known mixture. The error of such a procedure was estimated at 4 wt%.

The mean size of SiC crystallites was assessed by the Scherrer formula. As the basic formula assumes monodisperse spherical particles, we used a modified formula including a corrective factor¹²⁻¹³ to account for the real crystals:

$$L = \frac{k\lambda}{D \cos \theta} \quad (1)$$

L is the full-width half-maximum (FWHM) of the SiC peak, $\lambda=0.15406$ nm Cu-K α wavelength, k a constant determined to be 0.9 by comparison with crystallite size as observed by TEM and D the mean size of the SiC crystallites.

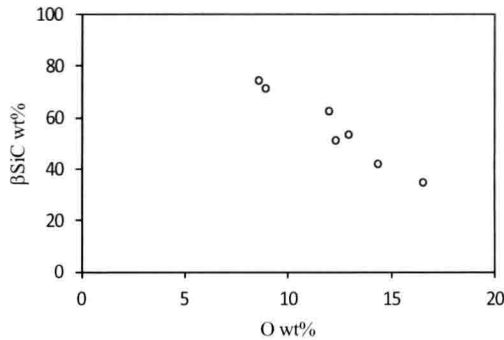


Figure 3: β-SiC crystallite size as a function of the oxygen wt%

The amount of β-SiC crystallites is clearly correlated to the oxygen content of the fiber: the higher the oxygen content, the less the amount of β-SiC crystallites (figure 3). This behavior is in accordance with the Si-C-O phase diagram. It was furthermore observed that the size of the β-SiC crystallites increases with their amount.

Determination of the Free Carbon Content

The amount of free carbon embedded into the continuum was deduced from the atomic composition of the fiber, according to the above described calculation. It was also measured by NMR ¹³C analysis. The recorded spectrum is composed of two broad peaks at ~19ppm and 119ppm which respectively correspond to C sp³ and C sp². From the proportion of each type of carbon hybridization, it is possible to estimate the amount of carbon bonded to silicon and the amount of free carbon.

The amount of free carbon measured by the two approaches are in good accordance (figure 4) and shows the same order among fibers. It can nevertheless be observed that amount of free carbon deduced from the measured atomic composition is always higher than the amount measured by NMR. As a general trend, carbon content of Tyranno fibers is much higher than that of Nicalon fibers, because of the type of polymer used as precursor. The maximum carbon content is measured in the Tyranno S fiber and the minimum in the Nicalon NLM207.

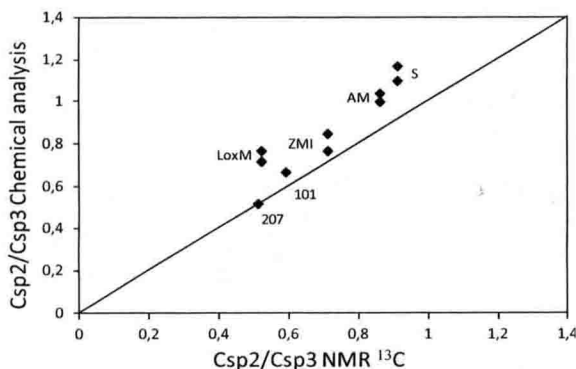


Figure 4: comparison between the ratios between free and tetrahedral carbon deduced from the measured atomic composition of the fibers and its measurement by NMR ^{13}C .

Fractographic Examination of Rupture Patterns

The mechanical properties of fibers are strongly dependent on their chemical composition. For instance, first generation Si-C-O fibers have a tensile modulus of about 200 GPa⁶, though the modulus of near stoichiometric fibers reaches almost 300-400 GPa¹⁴. The morphology of the rupture surface, which is typical of brittle materials, is indicative of the failure mechanism: mirror, mist or hackle-like and crack branching¹⁵⁻¹⁷. The rupture pattern and its boundaries were used to identify the size of the initial flaw²³⁻²⁴, assess toughness^{18,19}, strength¹⁹⁻²², or residual surface stress^{21,24}.

Single fiber tensile properties were measured on a micro-tensile testing machine at a constant loading rate of 0.5%.min⁻¹ using a gauge length of 25 mm. In order to obtain a good statistical analysis, 40 to 50 samples were tested for each type of fiber. The fiber diameter was measured by laser diffraction prior to testing, and was observed post mortem by SEM.

SEM Observations

SEM observations of fracture surfaces are aimed at identifying the crack origin, measuring the area of typical flaw features and checking the diameter of the fiber. Among the several hundreds of rupture surfaces that were observed by SEM, seven typical families of characteristic flaws were identified: five are originating from surface flaws and two from internal flaws. These characteristic flaws are shown in Figure 5. The A-type flaws have a well-known penny shape contour, the B flaws are small particles present at the surface of the fibers, the C flaws are small surface damages that may result from manipulation or contact between fibers, and the D flaws are chemical composition heterogeneities¹⁷. In the case of E-type flaws, though rupture clearly originates from the surface, the flaws cannot be observed by SEM because of their particularly small size or the poor image contrast. Concerning the rupture induced by internal flaws, the F flaws are internal inclusions²⁵ or voids and G

flaws are typical penny shaped cracks (similar to A). These latter flaws were only observed on Hi-Nicalon fibers. A statistical analysis of the flaws responsible for the fiber ruptures is summarized in Figure 6. When plotting, for each fiber, the proportion of ruptures originating from internal and surface flaws, the behavior of the Hi-Nicalon fiber is clearly different from that of the other fibers: almost all their observed ruptures originate from internal flaws. This result is in accordance with G. E. Youngblood et al.²⁵ observations stating that all of the Hi-Nicalon fiber ruptures originate from internal flaws, though J. Hurst²⁶ only observed 60% of ruptures induced by internal flaws. As a general trend, the proportion of Nicalon fiber ruptures induced by internal flaws is higher than that of Tyranno fibers, except for the Lox-M fiber.

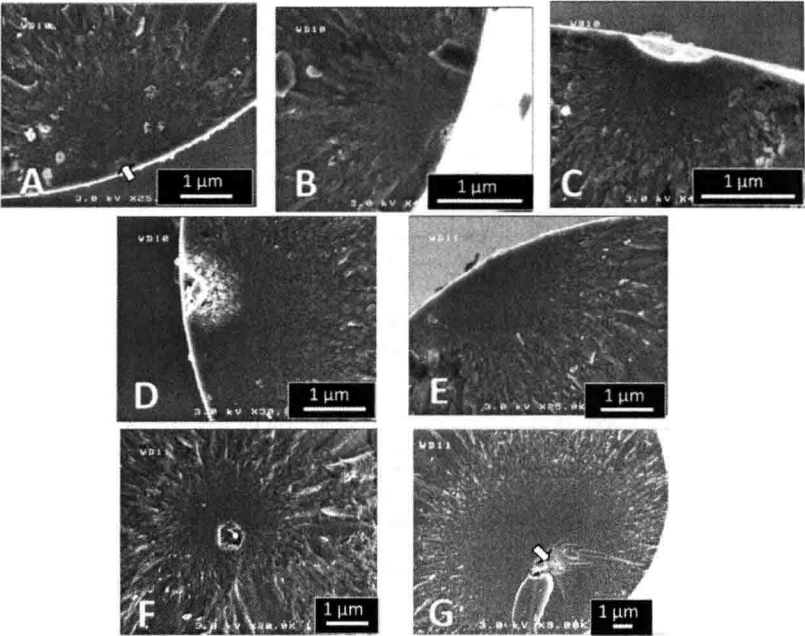


Figure 5: Classification of flaw pattern

Surface flaws: (A) penny shaped crack, (B) particle, (C) surface damage, (D) chemical heterogeneity, (E) no observable trace by SEM,
Internal flaws: (F) internal inclusion, (G) penny shaped crack