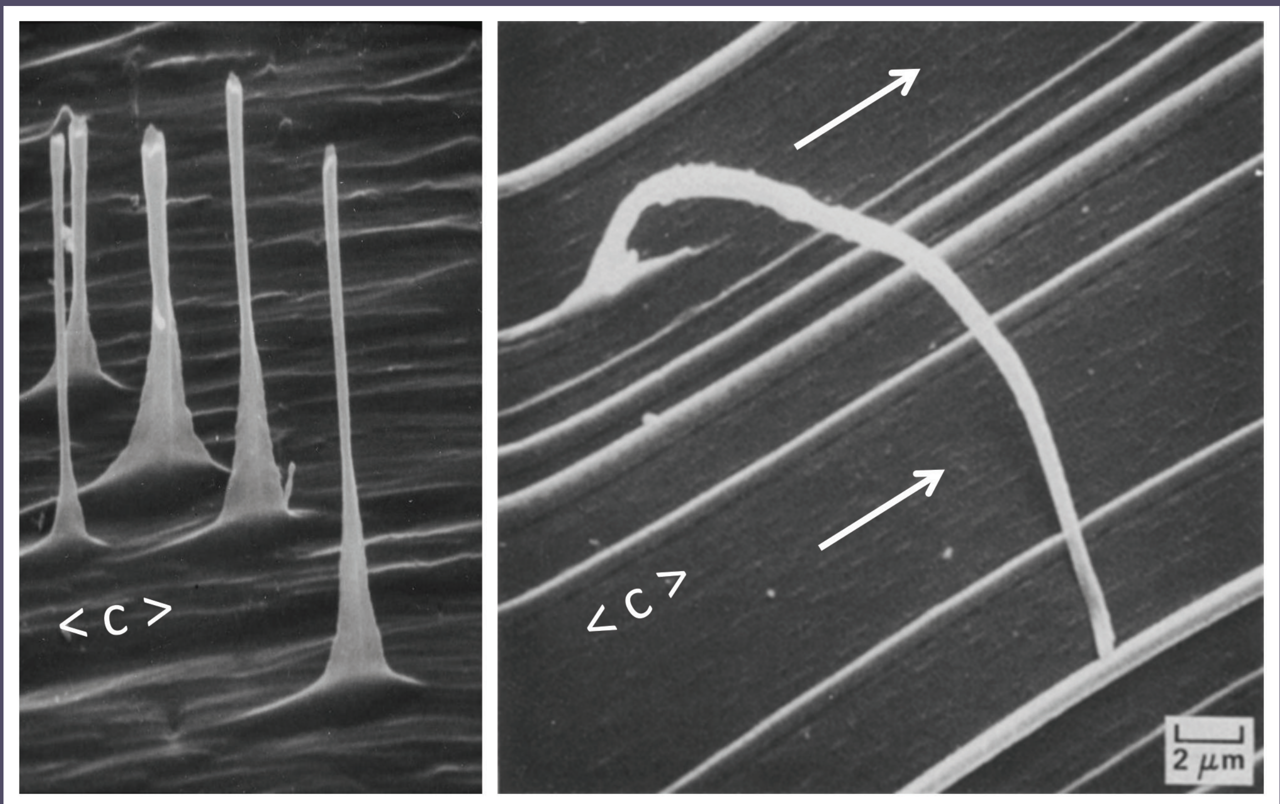
ENGINEERING PHYSICS OF HIGH-TEMPERATURE MATERIALS

METALS, ICE, ROCKS, AND CERAMICS



NIRMAL K. SINHA ▪ SHOMA SINHA

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Metals, Ice, Rocks, and Ceramics

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WILEY

This edition first published 2022

Registered Office
John Wiley & Sons, Inc., 111 River Street, Hoboken, NJ 07030, USA

Editorial Office
111 River Street, Hoboken, NJ 07030, USA

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Library of Congress Cataloging-in-Publication Data

Names: Sinha, Nirmal K. (Engineer), author. | Sinha, Shoma, author.
Title: Engineering physics of high-temperature materials : metals, ice, rocks, and ceramics / Nirmal K. Sinha and Shoma Sinha.
Description: Hoboken, NJ : Wiley, 2022. | Includes bibliographical references and index.
Identifiers: LCCN 2021027147 (print) | LCCN 2021027148 (ebook) | ISBN 9781119420484 (cloth) | ISBN 9781119420453 (adobe pdf) | ISBN 9781119420460 (epub)
Subjects: LCSH: Materials at high temperatures. | Deformations (Mechanics)
Classification: LCC TA418.24 .S56 2021 (print) | LCC TA418.24 (ebook) | DDC 620.1/1217–dc23
LC record available at https://lccn.loc.gov/2021027147
LC ebook record available at https://lccn.loc.gov/2021027148

Cover Design: Wiley
Cover Image: © Nirmal K. Sinha

Set in 9.5/12.5pt STIXTwoText by Straive, Pondicherry, India

10 9 8 7 6 5 4 3 2 1

The authors would like to dedicate this book to their loving grandson and nephew, Indro Neel Sinha. The proposal of this book was accepted by Wiley on the day he was born in December 2016.

Contents

Acknowledgments

Both authors would like to acknowledge the patience and support of their entire family – particularly Supti Sinha, Priya Sinha, and Roona Sinha. Without them, this work would not have been possible.

Nirmal Sinha: I am indebted to Dr. Arthur Carty, the then President of National Research Council (NRC) of Canada, for providing the opportunity and necessary funds in 1998 to build the required facilities within the Institute for Aerospace Research (IAR) of NRC for carrying out ice-based mechanical tests on various gas-turbine engine materials, relevant to this book and without which this book would not have been written. For about three decades prior to this, I had been encouraging materials scientists around the world to extend glass- and ice-based constitutive equations to high-temperature metallic alloys, but progress was limited until this opportunity.

This book presents progressive development that started from working on glass technology. The late Dr. Sydney Bateson (Director) and J.W. Hunt (Manager) of Glass and Ceramics Laboratory, Duplate Canada Ltd. (Oshawa, Canada), mentored, inspired and provided me with the freedom during 1964–1971 to conduct studies related to thermal tempering and strength improvement of window glasses. Duplate (acquired by Pittsburg Plate Glass), sole supplier to all the automobiles manufacturers in Canada (General Motors, Ford, and Chrysler), also allowed me to fulfil the requirements for my doctoral studies during 1967–1970 at the University of Waterloo. D. Tansley assisted me in designing and fabricating tempering and creep equipment in the laboratory at Duplate. A large number of persons, including C. McKnight in the manufacturing plant at Duplate, exposed me to the production problems and real-life issues in making automotive and structural safety glasses. Dr. K.M Baird of the Physics Division of NRC allowed me to borrow the first Canadian He–Ne gas LASER, which he had fabricated, for my rheo-optical studies on the tempering of glass, especially for the development of the scattered light technique for determining stresses in thermally toughened glass sheets. Dr. J.C. Thompson of the University of Waterloo helped me immensely in the preparation of my PhD thesis.

I am also indebted to the late Dr. Lorne Gold at the Division of Building Research (DBR) of NRC for the endless discussions, critical comments, and supply of his original experimental data on microcracking in pure ice, which enhanced the EDEV model for broad applications. The technical help of D. Wright, R. Jerome, and J. Neal for the years at the ice mechanics laboratory of DBR (NRC) is much appreciated. For the studies on superalloys at IAR (NRC), I am indebted to T. Terada, R.C. McKellar, and R. Kearsey for their excellent technical assistance, and to Dr. W. Wallace for the metallurgical discussions.

Prof. Brian Wilshire inspired me, in a very positive manner, to challenge many ideas popular in the high-temperature mechanics of metals and alloys that are also followed, often without question, in other materials science areas. It became an obsession to apply ice-based mechanics to metallic and other materials. This book may therefore be considered as complimentary to *Creep of Metals and Alloys* (Institute of Metals, 1985) by R.W. Evans and B. Wilshire.

For two decades of field observations at the world's largest skating rink (Rideau Canal Skateway, Ottawa, 7.8 km), and the experiments on the loading of floating ice described in Chapter 10, relevant to postglacial or global isostatic uplifting, I would like to acknowledge the technical help provided by D. Martin and his survey team, Design Division, Maintenance Division – Operations Group, and the Winterlude staff of the National Capital Commission (NCC) at Ottawa, including A.J. Capling, A.S. Fraser, M. Gauthier, and K. Tam.

Last, but most important, I am indebted to the Inuit communities of Pond Inlet (Baffin Island) for sharing their knowledge on sea ice as a material, and to various agencies of the Federal Government of Canada, namely Canadian Coast Guard (CCG), Atmospheric Environment Services (AES), Natural Resources Canada (NRCan), and the Canadian Armed Forces, for the logistical support in the High Arctic over a period of 30 years.

Shoma Sinha: Throughout my career, I have had the privilege to be supported and guided by many. Jeff Fraser from the NRC (Institute of Microstructural Sciences) not only kindled my interest in the fascinating world of materials and microscopy but his encouragement also gave me the confidence to pursue my interests. My doctoral research supervisor, Dr. Robert Wolkow, taught me to explore nature with a multi-disciplinary mindset. Dr. Gino DiLabio has helped guide me in critical thinking and planning – in both my research and overall career. I am also indebted to other members of my doctoral research team, including but not limited to, Dr. Stanislav Dogel, Dr. Jason Pitters, and Mark Salomans. The teams at Quantiam Technologies Inc., the Government of Alberta – Emerging Technology Industries and Queen's Partnerships and Innovation have further enabled me to explore and contribute to the multidisciplinary world of science and innovation.

My career would not be where it is today without the many amazing individuals who I know I can always rely on to be there with advice, mentorship, and friendship. The brilliant "brunch-ladies" and my co-founders of the WISER (Women in Science, Engineering and Research) Network – Gail Powley, Dr. Sharon Barker, and Dr. Heather Kaminsky – have created an amazing circle of support. I would also particularly like to acknowledge Heidi Au, Katherine Comber, Annette Dethlefsen, Mary-Ann LaFrance, Saurabh Ratti, Samia Sarkar, Emina Veletanlic, and Dr. David Waldbillig for always being there for me.

Engineering Physics of High-Temperature Materials

Preface

The development of knowledge in all branches of science and engineering has been so varied and rapid during the last century that it has become extremely difficult, if not impossible, for investigators to pay attention to different fields outside of their own expertise. As time progresses, each and every branch of scientific endeavor is getting subdivided and micro- divided, with specific jargon developing even within micro units, making it even more difficult to communicate with each other across specialties. The physics and engineering of high-temperature materials is one such special area, and yet it touches many fields in many ways.

There is an ever-growing number of human-made materials like ceramics, metallic alloys, and superalloys used specifically in high-temperature applications in areas such as the nuclear, chemical and aerospace industries. This may also include materials developed by design on the basis of nanotechnology and grain-boundary engineering for very specific uses. Then, there are rocks of geophysical interest (such as with respect to tectonics and post-glacial uplifting) existing at high temperatures within the depths of Earth and floating on magma, and ice (freshwater and saline sea ice) floating in its molten state in lakes and oceans. It would be impossible to cover all the complicated phenomena of different materials in a single book. However, the principal strengths of a book like the present one is the manner in which it covers many different materials all together. This could also be a weakness if descriptions are not clear enough to facilitate an understanding of the complicated physics and mechanics in widely differing materials. Some difficulties can be overcome by restricting topics relevant only to inorganic crystalline materials that would include the most abundant materials on Earth – ice and rocks, in addition to *manu-made* (gender-neutral term derived from *Manushya* in Sanskrit) and manufactured metallic-based engineering materials used in various industries such as aerospace, power generation, and nuclear technology. Further obstacles can be removed by concentrating on materials at or used at high homologous temperatures greater than about one-third of the melting point, T_m in Kelvin. In this manner, it is indeed possible to draw attention to a common string that unites most, if not all, *apparently different polycrystalline* materials and topics. Many time-honored, empirically derived relations will be explained on the basis of a simple, microstructure-sensitive, Elasto – Delayed-Elastic – Viscous (EDEV) model.

High-temperature materials science and engineering sounds like a specialized branch of applied science, but it can actually be considered as one of the most general areas of modern science and technology. This book is prepared with the intention of making it known that apparently dissimilar polycrystalline materials, such as metals, alloys, ice, rocks, and ceramics – and even glassy materials – behave in a very similar manner at high temperatures. This book, therefore, is aimed at a variety of experts, such as *metallurgists* to *metal physicists*, *glaciologists* to *ice engineers*, *solid-earth geophysics*, *earth scientists* to *volcanologists*, and *cryospheric* and *interdisciplinary climate scientists*. The critical question addressed is, what is really meant by "high temperature," and why? What is the microstructural-based rationale for defining high temperatures?

Materials scientists (materialogists) universally agree that temperatures, T, above about one-third of the melting point, T_m in degrees Kelvin, are *high*. For metals and alloys, it is unanimously recognized that $T > 0.4T_m$ is unquestionably categorized as high-temperature because intergranular cracks (called *wedge* or *w-type*) along the grain-boundaries (comparable to the size of grain facets) are predominantly observed at such temperatures, particularly in polycrystals. Grain-boundary spherical or elliptical voids (called *cavitation* or *r-type*) are also commonly noticed features in deformed or fractured materials. To this list of readily observable microstructural features, we consider a very special aspect of high-temperature deformation and failure processes – that, to-date, has not derived much attention from materialogists in general. It is the recoverable delayed elastic strain (des) in addition to elastic and viscous (matrix dislocation creep) deformation. For example, complex aerospace alloys exhibit a significant amount of delayed elastic effect not only during the primary or transient stages, but also during the tertiary creep regime. Progress made in ice mechanics, experimental as well as theoretical, have proved to be a fertile ground for explorations toward understanding the onset of interfacial failure processes in polycrystalline materials during the primary creep and eventual failures at high temperatures. The modern knowledge summarized in this book demonstrates that *delayed elastic strain* can be measured precisely at any stage of high-temperature deformation through the careful design of experimental techniques (e.g., Chapter 4). This is illustrated in Figure P.1.

As mentioned earlier, a constitutive model, named as the Elasto – Delayed-Elastic – Viscous (EDEV) model, was developed that recognizes delayed elasticity (that can be measured experimentally for quantitative verifications) as one of the most important aspects of high-temperature engineering materialogy. As this text will show, it has been demonstrated that delayed elastic strain plays crucial roles in governing every aspect of primary (often called *transient*) creep curves and engineering stress-strain diagrams and strain-rate-dependent strength (such as 0.2% offset yield and ultimate strength) properties. Finally, and very importantly, grain-facet size cracks are initiated during primary creep, when des reaches a critical stage (Chapters 5, 6). The kinetics of microcracking and crack-enhanced viscous (or dislocation) creep, essence of the EDEV model, leads to tertiary or accelerating stages in constant-stress creep or constant strain-rate deformation (Chapters 7, 8). The processes of grain-boundary shearing (often referred to as sliding in the literature) induce recoverable delayed elastic strains. The grain-boundary shearing mechanisms also govern the initial-strain (or initial-constrain) sensitivity of stress-relaxation (SR) at high homologous temperatures, as presented in Chapter 9. The crack-enhanced EDEV model, therefore, provides a physics-based elucidation for the phenomenological observations on a huge number of engineering materials. And the methodology is very simple. Material characteristics for creep, and the kinetics of grain-facet size cracking during creep, like those provided in Table 7.1 for ice, can be obtained for other materials by performing the appropriate strain relaxation and recovery test (SRRT) (Chapter 4), including the use of acoustic emission (AE) technology, and emphasizing, of course, evaluation of recoverable delayed elastic response.

Engineering design is most often based on "effective" elastic response, yield strength such as 0.1 or 0.2% offset yield stress, and/or design curves summarizing stress-time-temperature dependence of some specified strain. All these characteristics are strain-rate sensitive and have been shown to be governed by primary or transient creep at high temperatures. It is shown in this book that primary creep is linked strongly to observable and precisely

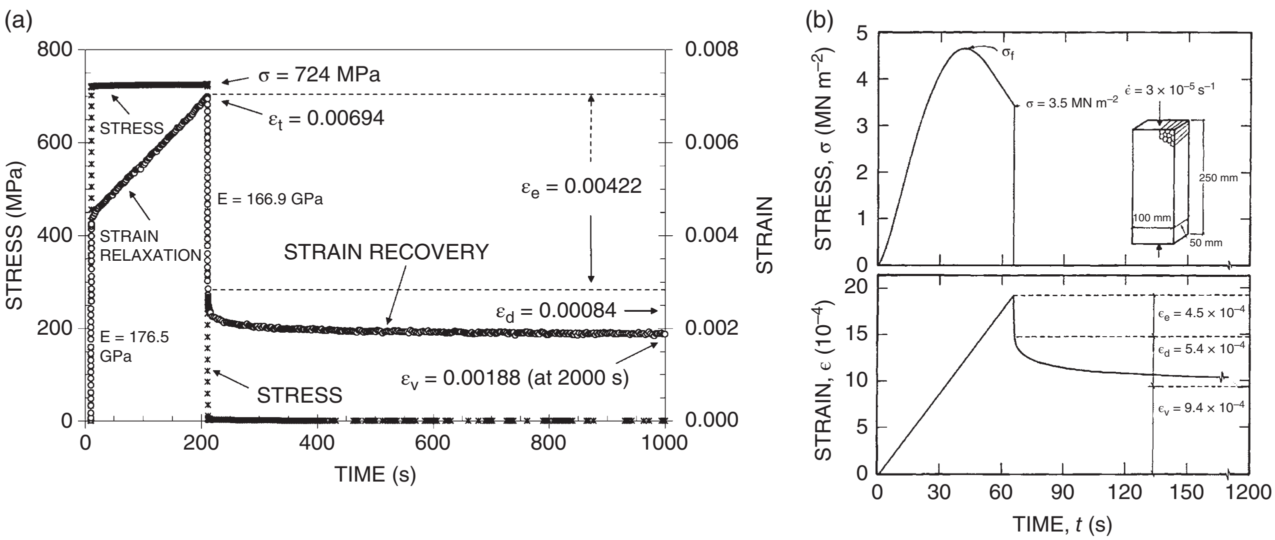

Figure P.1 Delayed elastic strain (des) recovery. (a) constant-stress creep of nickel-base Waspaloy forgings at 1005K and 724 MPa; (b) constant strain-rate strength test of directionally solidified (DS) ice at 263K ($0.96T_m$) and strain rate of $3 \times 10^{-5}\ s^{-1}$, as described in Chapters 4 and 6. Source: (a) N.K. Sinha, unpublished; (b) Sinha (1988a) with permission from Springer Nature.

quantifiable delayed elastic phenomena, and that it is of utmost importance not only for characterizing the propagation of seismic waves in rocks (well recognized by geophysicists and volcanologists), but also for the prediction of strain-rate-sensitive 0.2% offset yield strengths, extremely important for design engineers. This book fills this gap in materials science in a significant manner.

There are a number of excellent books published in the past with a primary emphasis on metals and alloys. These publications have received wide-ranging attention from metallurgists over the last 50 years or more. However, none of these well-known publications have (to the authors' knowledge) provided any information on *grain-size-dependent nucleation and the kinetics of grain-facet size microcracking activities and crack-enhanced matrix creep, which starts during early stages of primary (transient) creep, leading to minimum creep rates and tertiary stages*. Minimum creep rates are evolved properties and are in fact predictable. Minimum creep rate does not necessarily mean steady-state creep due only to the dynamics of dislocation creep/climb mechanisms. The use of the usual experimentally evaluated characteristics of the minimum creep rate as a fundamental material property was recognized as being inappropriate by several investigators, but this is still largely ignored. None of the available books that focus on metallurgical processes take notice of the fact that strain-rate-sensitive 0.2% yield stress depends on characteristics of transient creep. This yield stress is actually predictable for real engineering materials (e.g., Ni- or Ti-base superalloys used in gas turbine engines) on the basis of the EDEV model using material characteristics that can be obtained from independent SRRT tests (elaborated and substantiated in Section 5.16 of Chapter 5).

The preceding text summarizes fundamental concepts that, although duly acknowledged in different ways by materials experts in different fields, are yet to be addressed comprehensively in a text that ties it all together. Moreover, the implications of applying the knowledge to different fields is vast: from predicting/designing to account for the creep of nickel-base turbine blades in aerospace or power engineering, to guidelines for ice fishermen about how long a vehicle can remain parked on a floating ice sheet, or even to describe certain aspects of post-glacial uplift and plate tectonics, including man-made reservoir-induced earthquakes, known as reservoir-triggered seismicity (RTS).

The traditional concept of "strength" implies a *specific* material property. But *the strength* of a material is a low-homologous temperature concept, say, less than about $0.3T_m$. This low-temperature concept, based primarily on stress-strain diagrams without any reference to time, does not apply at the elevated temperatures relevant to all high-temperature engineering, for example, hot sections of gas turbine engines or nuclear and power-generation applications. Strength at elevated temperatures is rate sensitive and is therefore not a specific property. Nonetheless, the concept of strength as a specific property (a low-temperature concept) has retarded growth in the understanding of microstructure–property relationships and failure processes in engineering components in general. The application of this concept has misleading implications, drawing away from one basic fact: transient or primary creep stage, involving the initial periods of damage accumulation, plays a dominant and perhaps decisive role in many engineering problems. In Chapter 8, we will use the *crack-enhanced Elasto – Delayed-Elastic – Viscous (EDEV) model* for predicting the rate sensitivity of strength in a rational manner.

One of the primary intentions of writing this text is to draw attention to the fact that polycrystalline ice can be used as an "ideal analogue" material to explain certain peculiarities of the elevated temperature response of engineered as well as natural materials. One such peculiarity is the observation that a polycrystalline material may exhibit both ductility and brittleness in a simultaneous manner. And this may happen at rather low levels of stress for engineering applications. But again, what do we mean by low or high stresses appropriate for high temperatures? Only by examining and analyzing the initiation of grain-facet size cracks that can lead to tensile fracture can we offer a satisfactory mathematical and physical description for the stress as low or high. There is sufficient evidence to show that stresses higher than about $1 \times 10^{-5}\,E$ at $T > 0.4T_m$ (where E is the Young's modulus) may be considered as high stress for polycrystalline materials at high homologous temperature.

The onset of microcracking activities in pure, transparent ice can be monitored both visually and using AE technology. This dual process of evaluation is not possible for most opaque materials like metals, ceramics, and most rocks. Since it is not possible to visually identify the tiny grain-facet size cracks inside most engineering materials, including bubbly ice, one-to-one correspondence between AE or microseismic activity (MA) signals and cracks could never be made. This is the dilemma for all metallic and ceramic materials. The predicament due to the opacity of specimens in most engineering materials allows AE/MA signals, even with 3D locator systems, only for monitoring the overall crack-damage processes. We will discuss these issues in Chapter 4 (Section 4.10) and Chapters 7 and 8 for clarifying the advantages of using pure ice as an ideal analogue material for studies on engineering materials in general.

Although the very powerful Elasto – Delayed-Elastic – Viscous (EDEV) model, described and applied for a wide range of engineering applications (Chapters 6–10), was developed from the rheological investigations on glass (Section 5.5 in Chapter 5), we are still unaware of any satisfactory answer as to why glass exhibits delayed elasticity identical to polycrystalline ceramics, metals, and complex alloys (see Section 5.6, Chapter 5). Why is the deformation-induced birefringence (photoelastic effect) in glass independent of viscous strain and coupled "only" to pure elastic response of its complex structure?

Sea ice in the Arctic Ocean plays one of the most important roles in modifying the climate of the world. Sea ice in the Antarctic region is marginal and seasonal, as described in our earlier book, *Sea Ice: Physics and Remote Sensing* (AGU/Wiley, 2015). No doubt, we must pay attention to the formation and decay of sea ice as a measure of climate change. Coincidentally, air- or space-borne images of sea ice bear all the likeness of micrographs of metals, alloys, rocks, and ceramics, as pointed out in Chapter 11. Ice floes in the oceans can be characterized as grains in polycrystalline materials. On the other hand, an image of floating pack-ice may also evoke likeness to Earth's tectonic plates and sub-plates. Relative movements of sea ice floes with respect to each other and rafting can be described as divergence, convergence, subduction, etc. Can we apply the lessons learned from the bearing-capacity of floating ice, on the basis of the EDEV model, to large-scale global phenomena such as post-glacial uplifting (see Chapter 10), which is a very complex issue related to the convergence and subduction of plate tectonics or RTS (see Chapter 11)?

In Chapter 1, we introduce three major cooling vents for Earth as the "Trinity of Earth's Cryospheric Regions." *Cryosphere* is historically an accepted term, depicting cold (from the human point of view), ice-rich areas, including the atmosphere. Thermomechanically speaking, ice can be considered as cold only if the temperature is significantly below $0.4T_m$ or 109K (−165°C). Earth's cryosphere, therefore, consists of three relatively *hot zones*: primary (two polar areas) and secondary (the Alps, Andes, Himalayas, Rockies, etc.). The *Trishul* or Trinity of major cryospheric zones of the world is: the North and South poles, and the Himalayan belt in the middle (Figure P.2).

Figure P.2 "Trishul" (trident) of the two primary – North (N) and South (S) – polar regions, and the secondary regions represented by the Himalayas (H), with concentrations of snow and ice at extremely high homologous temperatures. Source: Visionary sketch by N.K. Sinha.

Materialogists would perhaps give limited thought to the geophysically established fact that the secondary cryospheric zones of Earth – the Himalayas, Andes, Rockies, etc. – are products of high-temperature phenomena active deep underneath Earth's crust. Plate tectonics, very similar to sea ice dynamics, is briefly presented in Chapter 11. It is shown that the zone of reservoir-induced earthquakes (or RTS), such as the Koyna–Warna area in India, may be predicted on the basis of the Elasto – Delayed-Elastic (EDE) aspect of the EDEV equation.

History based on engineering physics looks to be the domain of professionals in metallurgy and materials science or materialogists. Where so much of the past, even the chronology, has to be teased from articulated intellectual objects emphasized in textbooks, scientific papers, and monographs, there surely must be need for a new perspective. However, much of the information required with state-of-art experimental observations was missing. The principal author, in particular, decided therefore to take a path that was a deviation from the normal.

Nirmal K. Sinha and Shoma Sinha

1

Importance of a Unified Model of High-Temperature Material Behavior

CHAPTER MENU

1.1 The World's Kitchens – The Innovation Centers for Materials Development

Engineering development is intricately linked with our understanding and manipulation of various kinds of materials, which are either readily available on land and sea or fabricated from them. Long before the dawn of civilization, Earth's surface had gone through many cycles of freezing and thawing. The ice age and deglacierization or melting of glaciers and ice sheets, still persisting on Earth's surface, played a pivoting role in shaping our lives and materials development. There is no question that ice played an important role in shaping the land and making adjustments in living conditions. But what does ice have to do with a book like this, entitled *Engineering Physics of High-Temperature Materials*? In this book, we demonstrate how the knowledge of the physics of ice – a material that exists close to its melting point – can improve our understanding of all high-temperature engineering materials. However, let us first explore a bit of human history and development in the usage of building materials.

The "Stone Age," an archeological term of the three-age system, was characterized by the use of stone as implements and ended variably between about 9000 and 2000 BCE in different areas of the world with the development of metalworking. It has been divided into the Paleolithic, Mesolithic, and Neolithic periods (Bates and Jackson 1980). The "Bronze Age," characterized by metalworking and primarily the alloying of copper with tin and arsenic, took over between roughly 3000 and 1000 BCE with the "Iron Age" starting roughly between 1200 and 600 BCE.

The advent of the current cycle of global warming, which roughly began around 18 000 years ago (estimated peak period of the last glaciation), led humans as well as other

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species to move into areas previously covered with ice. A straightforward, easy-to-read introduction to various aspects of geology, which is useful to materials scientists and engineers who are not familiar with geological science, can be found in *Physical Geology* (Plummer and McGeary 1985). The process of deglaciation took a long time. The bulk of the ice sheets on Earth's surface melted away around 10 000 years ago. The global sea level was at its lowest level during the peak period of the last ice age, but it started to rise as the meltwater returned to the oceans. However, as ice melted, the ice load on the glaciated land, floating on Earth's molten mantle, decreased and the land areas started to rebound rapidly in the beginning and then slowed down. Most of the global land areas being used today, including those far away from the previously glaciated areas, became available about 6000 years ago, with one well-documented exception of the loss of land – Australia's Great Barrier Reef (GBR). The 2400 km long GBR did not exist 15 000 years ago.

We may safely say that materials development accelerated from the middle of the current period of geologic time, which is known as the "Holocene Epoch" that started around 12 000 years ago. The Holocene (meaning wholly or entirely new) period began at the close of the Paleolithic Ice Age. Here, the "Age" (geochrone) is understood as the time interval during which a particular event occurred or a time characterized by unusual physical conditions, which is one of the many definitions used for describing a unit of geologic time (Bates and Jackson 1980; Lapidus 1987).

Trinity of Fire
PRODUCTION
CONTROL
MAINTENANCE

In the early Holocene age, the primary human needs arose from requiring protection from the elements and broadening food sources. These necessities led to the quest for controlled fire and with it the realization of the need for techniques for producing (P) and controlling (C) fire at will, rather than depending on its natural sources, as well as maintaining (M) fire as the need arose.

The trinity of fire (PCM) – i.e. generating fire whenever the need arises, using it, and preserving the source – was probably the most important aspect of human development. PCM provided hominines with the opportunity to extend their habitats to colder regions of the earth where caves could be used as heated (air-conditioned!) and protected shelters. It also started a new era – the taste for cooked food and the beginnings of the kitchen for such food items anywhere, any time. The concept of "home cooking" and the advent of kitchens, marked, albeit debatable, the departure point from the cohabiting animal, although many animals and birds have developed the taste for burnt food produced during natural fires in forests or grassy lands.

Producing fire under dry environmental conditions was perhaps the beginning of this phase of human development and that of "kitchen" and "home cooking." PCM heralded a transition from nomadic, constantly moving communities of hunters and gatherers and cave dwellers, to relatively stable societies with fabricated housing, making tools for various applications, and eventually to the development of agriculture and thus creating new social structures, to name a few.

Trinity of Civilization
TECHNOLOGY
FIRE
LANGUAGE

No doubt, PCM was responsible for opening an unlimited number of opportunities for the development of wide-ranging technologies. As suggested by Gowlett (2016), the combination of technology (T), fire (F), and language (L), "can be seen as 'the big three', deeply connected [concepts] in the end and perhaps at earlier stages" of civilization. We may refer to this as the trinity of civilization.

The latest cycle of long-term global warming started around 18 000 years ago, which is the estimated peak period of the last glaciation. Of course, there were mini cycles of freezing and thawing within the Holocene age. The current stage of post-glacial period also helped the human race, equipped with PCM and TFL knowhow, to spread its wings to northern countries of the globe. As the ice cover melted and the flora and the fauna took over the arid landmass, migration of human race occurred in Scandinavia and Siberia in Eurasia and the continents of North America, as well as South America and the south-Pacific islands, and probably caused migration from the north and the center of the continent of Australia to its southern areas. The cyclic nature of the global-scale warming and deglaciation also forced the human race to relocate from time to time due to the rise of the oceans. There are legends that tell the stories about the First Nations of Australia moving inland as the GBR developed. The GBR did not exist during the Pleistocene (meaning most new or newer) epoch or more than about 15 000 years ago. The Holocene era is marked as the current age of global warming and deglaciation. During the latter part of the Holocene, repeated and "sustained" flooding due (most probably) to meltwaters from the Himalayan ice caps forced the Harappans of the Indus Valley Culture (IVC) to abandon their network of planned cities built with "modern-day" fired bricks along the Indus River.

Figure 1.1 Harappan style of brick production from clay. (a) Process of sun drying of clay bricks (in Kolkata) Source: Courtesy of Srewoshi Sinha; (b) and (c) Fired products at a construction site in New Delhi in 2010. Source: Nirmal Sinha.

The spread of knowledge for producing fire under widely different environmental conditions to various communities *living under* diverse environmental conditions evolved over thousands of years. All along the development of settlements of humankind – whether in the well-known cradle of civilization or cultures in Egypt, Mesopotamia, or the not so known, but still living, IVC of Harappa and Mohenjo-daro in ancient India (National Geographic Society 1979; Keay 2004) – kitchens with fireplaces provided the place for innovative thoughts on making "ceramic" pots and pans and building materials from baked clay. Interestingly, Harappan bricks were used for making railway lines even a hundred years ago, and the IVC technology in its most rudimentary stage (handmade, sun-dried, and fired in stacks) is still practiced and it provides millions of jobs all over the Indian subcontinent, making bricks and constructing even high-rise living quarters (Figure 1.1).

Innovation naturally led toward the use of various silicate materials for making sun-dried bricks, as well as pots and pans for storing dried food and grains. It is strongly believed that cooking over fires using sun-dried pots led to the invention of durable fired clay or terra-cotta pots for storage of water as well. In ancient kitchens, the containment of fire by sun-dried clay blocks, made from fine-grained clay materials, probably led to the observations on high-temperature sintering of clay particles. The next stage was the development of fired bricks for making shelters. The challenge was to make bricks without cracks.

Building materials, whether they are rocks or metals, are mostly polycrystalline materials. Glass, unlike crystalline materials, is amorphous and lacks any periodicity in arrangements of its atomic structure. The world of natural silicates is vast, and molten silicates are part of the magma deep inside Earth's surface. If a mixture of natural silicates melts and then cools rapidly below its melting point, a silicate glass is formed. Naturally, on top of Earth's surface, there are localized glassy phases within volcanic rocks. No wonder glass became one of the oldest and familiar materials known to mankind and found use in many forms.

Glass as we know it – generally in the form of containers, sheets, mirrors, etc. and in buildings as cladding and window materials – is primarily the form of silicate glasses, particularly soda-lime–silica glasses. There is a wide variety of optical, cookware, and specialty glasses. Very rarely, unless laminated with layers of polymers, glass is used for any load-bearing components of buildings. Glass plates or sheets develop microscopic surface flaws after manufacturing. Brittle propagation of these surface cracks is the limiting factor for the use of glass. Creep properties of window glass, even for laminated products, limits its use at high temperatures. Understanding the rheological properties of glass, however, is crucial for manufacturing glass articles and strengthening them for use as safety glasses and tempered windows of automobiles and buildings. We will broaden the exploration of the structural aspects of glass in Chapter 2 and experimental techniques for microstructural analysis of ice (especially dislocations as line defects and pileups) in Chapter 3. Rheology, failure of glass, and thermal tempering toughening are presented in detail in Chapters 4 and 5. The mechanisms for the nucleation of grain-facet-sized intergranular cracks, kinetics of crack generations, and crack-enhanced creep and failures are covered extensively in Chapters 7 and 8.

Glass is amorphous in structure, but most natural and manufactured metal-based engineering materials are polycrystalline materials. However, a rheological model, called Elasto – Delayed-Elastic – Viscous (EDEV) model developed originally for stabilized glasses at elevated temperatures (Sinha 1971), can be, and has been, used as the foundation for developing grain-size-dependent EDEV models for high-temperature creep and fractures of pure polycrystalline metals and complex alloys, ceramics, and rocks. The delayed elastic effect and its recoverable aspects have been linked to grain-boundary shearing processes. The connectivity to engineering materials, such as complex titanium-base and nickel-base superalloys used in gas turbine engines for power generation and in the aerospace industry, is based on the physics of the grain-boundary areas. The history of this scientific and technical development is fascinating and has been described in Chapters 5–9. The description covers the whole range from phenomenological to microstructure-based, grain-structure-sensitive EDEV modeling for deformation, cavitation, dilation, and strength. The predictive power of this EDEV model has been demonstrated by explaining the ductile-to-brittle transition and success of the classical and ever popular minimum creep-rate-based models (e.g. the Monkman–Grant (MG) relationship) for failure or fracture. EDEV model is also capable of quantitatively predicting the strain-rate sensitivity of 0.02% yield and upper-yield (ultimate) strength, as well as relaxation of stresses under constraint conditions. The possible extension of this EDEV model to get an understanding of the post-glacial uplifting is covered in Chapter 10 while the hypothetical uses of the EDEV model for understanding certain issues with plate tectonics are presented in Chapter 11.

1.1.1 Defining High Temperature Based on Cracking Characteristics

For any load-bearing engineering component, the propagation of inherent flaws or cracks at the surfaces or in the bulk and/or freshly nucleated microcracks and their multiplication under load is the most challenging factor to design for. At high temperatures, materials also exhibit time-dependent deformation or creep. Such rheological properties, including the kinetics of nucleation, growth, and propagation of microcracks, and their size and shapes with respect to the crystalline structure, are essentially the limiting factors that control the usage of these materials. Of course, the design of most engineering components is also limited by the amount of creep deformation. The question is – How can we define *high temperature* in a rational manner?

Trinity of Cracks
TRANSGRANULAR
INTRAGRANULAR
INTERGRANULAR

In order to define a "high temperature," let us consider the general characteristics of cracks seen in polycrystalline materials during deformation and after failure in a wide range of temperatures. Experimentally observed microstructural characteristics of load-induced cracks in a wide variety of polycrystalline materials can be divided into three basic types – transgranular (across grains), intragranular (within grains), and intergranular (along grain boundaries). It is particularly insightful to consider the characteristics of cracks within a material by considering the operating temperature with respect to the materials' specific melting point, T_m, in kelvin. Transgranular cracks are dominant at low temperatures ($<0.3\ T_m$). Intergranular, grain-facet-sized cracks readily form at temperatures higher than $0.4\ T_m$.

Thus, the simplest thermodynamically based and fracture characteristic inspired definition of high temperature for a polycrystalline material is the regime of intergranular cracks at operational temperature higher than a third of the melting point: higher than $0.3–0.4\ T_m$. In addition to this general fracture-appearance-based definition of high temperature, throughout the text, we will add another, and perhaps the most important, aspect of the term "high temperature," based on the time-dependent recoverable components of deformation characteristics: delayed elasticity.

It is appropriate to mention here that turbine blades used in aeroengines are exposed to temperatures as high as $0.8\ T_m$ and the design engineer's goal is to push the operating temperatures even higher. At present, single crystals of nickel-base superalloys, without any grain boundaries (to avoid/minimize intergranular cracks), could take the challenge.

The above generalization of the "trinity of cracks" based on crack types, however, does not apply to amorphous materials with microheterogeneous (mechanically and chemically) structure, like natural silicates and complex inorganic glasses used in engineering applications. Unless there are inclusions trapped within the mass, glass objects fail primarily due to the propagation of surface cracks, which are either inherent following fabrications or nucleated during service life.

Glass is one of the oldest materials known to mankind. Glassy objects can be found in nature within the mass of a large variety of silicates. It is rather difficult to say what came first as consumable building materials for mass use: glass or ceramics (including fired bricks)? Actually, it is the glassy phase that gives the strength to sun-dried clay blocks after prolonged firing. The knowledge of making relatively crack-free fired (sintered) earthenware for storage of food and for cooking probably heralded the dawn of manufacturing fired bricks for mass consumption. All over the world, there are examples of early use of sun-dried bricks in constructing houses, but fired bricks probably led to the building of cities. Unique among them were the planned and designed Harappan-style settlements and cities along the Indus River in the Indian subcontinent. Residential quarters, identical to modern row houses, were constructed in these cities with prismatic fired bricks having rectangular cross-sections. The dimensions of bricks used in Harappan cities were also standardized (Keay 2004). So were the procedures for making them using clay and wooden frames and then by drying them in the sun, stacking them in the form of pyramids, and firing them with wood, charcoal, or coal dust. Even today, one can see this ancient technology at work all over India. One does not have to imagine the different stages of the manufacturing process for making fired bricks in ancient times. Figure 1.1 illustrates how the Harappan technology for mass production of fired bricks from clay, including the quality of bricks and even manual labor involved in handling the bricks at construction sites of multistory buildings, has remained unchanged for millennia.

The PCM of fire also led the human species to invent techniques for extraction of metals. High-temperature technology was also extended to the development of manufacturing tools for hunting and gathering from using tin and copper to making articles of bronze and then of iron and steel. With this knowledge came the development of high-temperature metallic alloys and it can also be linked to the "kitchen." During the early 1900s, cobalt-based and cobalt–chromium–tungsten systems were developed to make cutlery in addition to machine tools and wear-resistant hard-facing applications (Morral 1968; Beltran 1987). Incidentally, the knowledge gained from the metallurgical processes of making tungsten filaments for incandescent light bulbs and simple alloys for making coils inside elements for electric stoves and ovens led to the beginning of the development of complex high-temperature cobalt-based, nickel-based, and iron-rich alloys. The simple nickel–chromium alloy, discussed in the following text, heralded a new era of the use of superalloys.

Making earthenware marked the beginning of the world of engineering physics of ceramics. These days, we rarely use earthenware for cooking, but high-temperature ceramics are common in most of today's stoves, ranges, and cookware used in kitchens. Kitchens without bowls, jars, and bottles of glass are unthinkable. Today, superalloys are closely linked to the aerospace industry. In aircraft and land-based gas turbine engines, superalloys are used extensively. About 60%, by weight, of most modern gas turbine engine structural components, such as blades, vanes, and integral wheels, are made of nickel-base superalloys. Although air travel by jet aircraft is a common mode of transportation today, how many travelers can imagine that the reliability of modern jet engines based on superalloy technology is connected to the kitchens of the world. The development of nickel-base superalloys, actually, started almost 100 years ago due to the demands, among others, of the kitchens of the world. It started from the development of 20 wt.% Cr in an 80 wt.% Ni solid solution alloy for electrical heating elements (Betteridge and Heslop 1974; Ross and Sims 1987; Stephens 1989). Chromium was added to nickel to improve its strength and oxidation resistance.

During the 1940–1960 period, the most widely used precipitation hardened nickel-base alloys, belonging to the cubic system, were developed in the metallurgical laboratories for the hottest components of jet and rocket engines. In Chapter 2, chemical compositions of a few nickel-base superalloys are given to provide a quick glance at the world of gas turbine engine materials. During the decade of 1960–1970, directionally solidified (DS) nickel-base superalloys were developed for fabricating turbine blades used in modern gas turbine engines (Duhl 1987). Directionally solidified polycrystalline superalloys (belonging to the cubic system) are highly anisotropic, strong, and creep resistant. The long axis of turbine blades is parallel to the long axis of the columnar grains with large cross-sectional areas. The <001> axis is parallel to the long axis of the grains. The Hall–Petch relation for strength giving preference to fine-grained materials at low homologous temperatures is not applicable at elevated temperatures; large-grained materials are used at high temperatures. Grain boundaries are the sources of weakness at high temperatures. Single crystals of nickel-base alloys are now used for manufacturing turbine blades in today's advanced jet engines (Duhl 1989). Creep, fracture, and creep modeling of nickel-base single crystals are presented in detail in Chapter 5.

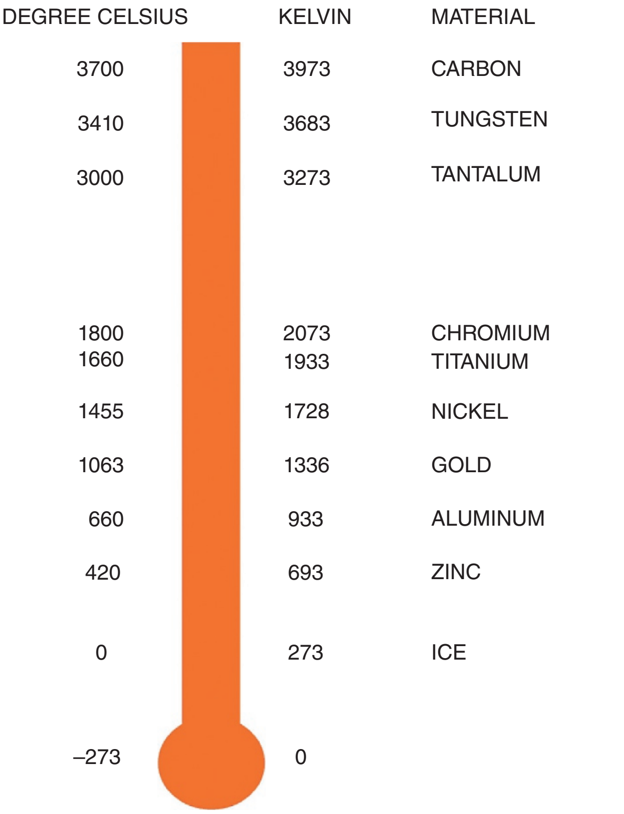

Figure 1.2 Melting points of some pure metals and ice.

It is highly possible that most metallurgists, materials scientists, and engineers may not be aware of the reality that "Mother Earth" produces three distinct types of DS columnar-grained polycrystalline ice every winter in freshwater lakes and vast oceanic surfaces of the earth. Natural ice, an oxide of hydrogen (H_2O), belongs to the hexagonal system (see Chapter 3). Although, granular snow–ice is commonly observed in thin surface layers of river, lake, and sea ice covers, as well as in glaciers and ice caps (Paterson 1994; Schulson and Duval 2009), engineering properties of this type of ice are not of significance because vast reservoirs of floating ice in lakes, rivers, and oceans are mostly columnar-grained, DS type. Two of these DS ice types have <0001> or <c> axis, of the columnar crystals either (i) parallel to the long axis of the grains, (ii) randomly oriented in the plane normal to length of the grains (mostly for freshwater lake and river ice). These two types of freshwater DS ice (types i and ii) are classified as S1 and S2, respectively (Michel 1978). Michel (1978) was the first person who discussed the physics of the creep and fracture of ice, their interconnectivity, and the relationship between its structure and mechanical properties for a wide range of engineering applications. A third type of DS ice (type iii), classified now as S3, has <0001> or <c> axis clustered in the plane normal to the long axis of the crystals (Weeks and Gow 1978, 1980). Floating sea ice mostly belongs to the S3 type (Shokr and Sinha 2015). Thus, the physics of fabricating DS types of superalloys can be linked directly to the knowledge gained from the physics of columnar-grained ice that naturally grows in lakes and rivers, as well as in salty water of the oceans (Dorsey 1940; Assur 1958; Gold 1965; Pounder 1965; Shokr and Sinha 2015). However, this nature-based lesson is not discussed in metallurgical books or publications (Duhl 1987; Ross and Sims 1987) dealing with the fabrication of DS-type superalloys.

An extremely important, yet startling, fact that leads some toward the philosophical issue of how humans (including even scientists and engineers) interpret temperature is the concept of ice as a high-temperature material. Although the "cryosphere" of the earth is derived from "cryogenics" (Greek: *kruos* frost, −*genes* born) and is understood universally as snow- and ice-covered areas, it should be realized that natural ice exists at temperatures extremely close to its melting point and is, therefore, the hottest material on the surface of the earth. Even an extremely cold body of ice at −40 °C (233 K) is equivalent to a very high temperature of 0.85 T_m (T_m = 273 K). This is still a dream operating temperature for gas turbine engine designers. Today, nickel-base superalloys, in the form of DS material and single crystals, are used exclusively for making blades and vanes for the hottest turbine sections of modern jet engines. The operating temperatures are restricted to a maximum temperature of about 1500 K, which is only about 0.8 T_m of the base material, i.e. nickel (see Figure 1.2).

The perception of ice as a cryogenic material, i.e. a material at very low temperatures, comes not from the thermodynamic point of view, but from the very fact that the temperatures associated with ice are uncomfortably cold for humans. The notion that ice is cold is enhanced by the very fact that it exists at "negative" temperatures in Celsius. This negativity is deeply rooted even in scientific minds of material scientists even though they are familiar with temperatures in kelvin and thermodynamic "homologous" scale.

It is not surprising to incline toward the popular cold-ice notion and treat snow/ice/permafrost-related engineering as a synonym for "cold regions engineering." There are several research and development laboratories in the world, dealing with issues related to glaciers, snow, snow avalanches, ice, and permafrost, and therefore these are named accordingly. For example, in Japan, there is the Institute of Low Temperature Science founded in 1941 in Sapporo. In the United States of America (USA), the US Army's Cold Regions Research and Engineering Laboratory (CRREL) was established in 1961 in Hanover, NH,