

A Study of the Effect of Thread Forming on the Susceptibility of Precipitation Hardened Ni-Based Alloy Fasteners to Hydrogen Embrittlement

API TECHNICAL REPORT 21D
FIRST EDITION, AUGUST 2024



American
Petroleum
Institute

Special Notes

API publications necessarily address problems of a general nature. With respect to particular circumstances, local, state, and federal laws and regulations should be reviewed. The use of API publications is voluntary. In some cases, third parties or authorities having jurisdiction may choose to incorporate API standards by reference and may mandate compliance.

Neither API nor any of API's employees, subcontractors, consultants, committees, or other assignees make any warranty or representation, either express or implied, with respect to the accuracy, completeness, or usefulness of the information contained herein, or assume any liability or responsibility for any use, or the results of such use, of any information or process disclosed in this publication. Neither API nor any of API's employees, subcontractors, consultants, or other assignees represent that use of this publication would not infringe upon privately owned rights.

API publications may be used by anyone desiring to do so. Every effort has been made by the Institute to assure the accuracy and reliability of the data contained in them; however, the Institute makes no representation, warranty, or guarantee in connection with this publication and hereby expressly disclaims any liability or responsibility for loss or damage resulting from its use or for the violation of any authorities having jurisdiction with which this publication may conflict.

API publications are published to facilitate the broad availability of proven, sound engineering and operating practices. These publications are not intended to obviate the need for applying sound engineering judgment regarding when and where these publications should be used. The formulation and publication of API publications is not intended in any way to inhibit anyone from using any other practices.

Any manufacturer marking equipment or materials in conformance with the marking requirements of an API standard is solely responsible for complying with all the applicable requirements of that standard. API does not represent, warrant, or guarantee that such products do in fact conform to the applicable API standard.

All rights reserved. No part of this work may be reproduced, translated, stored in a retrieval system, or transmitted by any means, electronic, mechanical, photocopying, recording, or otherwise, without prior written permission from the publisher. Contact the Publisher, API Publishing Services, 200 Massachusetts Avenue, NW, Suite 1100, Washington, DC 20001-5571.

Foreword

Nothing contained in any API publication is to be construed as granting any right, by implication or otherwise, for the manufacture, sale, or use of any method, apparatus, or product covered by letters patent. Neither should anything contained in the publication be construed as insuring anyone against liability for infringement of letters patent.

The verbal forms used to express the provisions in this document are as follows.

Shall: As used in a standard, “shall” denotes a minimum requirement to conform to the standard.

Should: As used in a standard, “should” denotes a recommendation or that which is advised but not required to conform to the standard.

May: As used in a standard, “may” denotes a course of action permissible within the limits of a standard.

Can: As used in a standard, “can” denotes a statement of possibility or capability.

This document was produced under API standardization procedures that ensure appropriate notification and participation in the developmental process and is designated as an API standard. Questions concerning the interpretation of the content of this publication or comments and questions concerning the procedures under which this publication was developed should be directed in writing to the Director of Standards, American Petroleum Institute, 200 Massachusetts Avenue, Suite 1100, Washington, DC 20001. Requests for permission to reproduce or translate all or any part of the material published herein should also be addressed to the director.

Generally, API standards are reviewed and revised, reaffirmed, or withdrawn at least every five years. A one-time extension of up to two years may be added to this review cycle. Status of the publication can be ascertained from the API Standards Department, telephone (202) 682-8000. A catalog of API publications and materials is published annually by API, 200 Massachusetts Avenue, Suite 1100, Washington, DC 20001.

Suggested revisions are invited and should be submitted to the Standards Department, API, 200 Massachusetts Avenue, Suite 1100, Washington, DC 20001, standards@api.org.

Contents

	Page
1	Scope..... 1
2	Normative References 1
3	Terms, Definitions, and Abbreviations..... 1
3.1	Terms and Definitions 1
3.2	Abbreviations 1
4	Test Plan 2
4.1	Test Protocols and Environment 2
4.2	Test Materials..... 3
4.3	Specimen Preparation 4
4.4	Incremental Step Load Test Procedure..... 7
5	Testing Results 8
5.1	Hardness Testing Results 8
5.2	EHE Testing Results 12
6	Discussion and Observations 12
6.1	Observations on Hardness Testing 12
6.2	Observations on Critical Stress Intensity 13
6.3	Observations on Fracture Strength Ratios 13
6.4	Consideration of Fracture Mechanics for HISC Susceptibility Assessment of Fasteners 14
6.5	Observations on Alloy 725–120k 17
7	Conclusions and Recommendations 20
7.1	Hardness Testing Conclusions..... 20
7.2	Fracture Toughness and ISL Conclusions 20
	Annex A (informative) Summary of Fracture Mechanics and Background of DTI and Hsr Equations 22
	Annex B (informative) Practical Application and Example Calculations of DTI and Hsr 27
	Bibliography 31

Figures

1	Example of Specimen Extraction Locations from Threaded Product..... 5
2	Example of the Thread Root and Thread Tip Microhardness Traverses Performed..... 6
3	Smooth-sided Threaded Specimen (left) Versus a Side-groove Constrained Threaded Specimen (right) After Testing (25x Magnification)..... 7
4	Image of a Full Side-groove Constrained Specimen After Testing (3x Magnification) 7
5	Example of CTOD Testing of SEN(B) Specimens to Determine Fracture Toughness of Material 8
6	Vickers (HV) Microhardness Testing of Cut Threads for Each Alloy 10
7	Vickers (HV) Microhardness Testing of Rolled Threads for Each Alloy 11
8	Results of Stress Intensities for Base Alloys and for Cut and Rolled Threads..... 13
9	Percentage FFS of Alloys Tested..... 14
10	Comparison of the $DTI_{\rho-EHE}$ Parameter for Each Alloy with Both Cut and Rolled Threads 16

Contents

	Page
11	Comparison of the Hsr Parameter for Each Alloy with Both Cut and Rolled Threads 17
12	Cross-section Microstructure of Alloy 725–120k (2020), Including Magnification of Rolled Threads 18
13	Cross-section Microstructure of Alloy 725–120k (2022), Including Magnification of Rolled Threads 18
14	Comparison of Microstructure at Center of Threaded Bar (T/2 Location) for Alloy 725–120k (2020) (top) and Alloy 725–120k (2022) (bottom)..... 19
A.1	“ σ ” and “W” Dimensions for a Typical SEN(B) Specimen 23
B.1	Thread Fracture Susceptibility (TFS) Diagram for a 12-UNR Threaded Fastener Under Simulated CP.... 30

Tables

1	Mechanical Properties of Tested Materials 3
2	Chemical Composition (wt. %) of Tested Materials..... 4
3	Average Vickers (HV) Microhardness Results for Cut and Rolled Threads..... 8
4	Results of Fracture Toughness Testing of All Alloys with Both Cut and Rolled Threads 12
5	Calculated DTI and Hsr Parameters for All Alloys with Both Cut and Rolled Threads..... 15
6	Chemical Composition Comparisons of 725–120k (2020) and 725–120k (2022) 20

Introduction

This study was completed in response to recommendations by API to investigate and quantify the relative hardness of threaded precipitation hardened nickel-based alloys (PHNAs), by both machined and cold rolled methods. This study also aligns with recommendations issued by the API Multi Segment Task Group on Bolting Failures (February 29, 2016), where it was concluded that “Product subcommittees should review and consider ... resolving existing conflicting properties specified in product specifications ...”. The information in this technical report has not been readily available as existing public or industry data.

Industry specifications often require hardness testing of PHNAs used for bolting in their final process condition, i.e. after all heat treatments, secondary processing, and machining. Threads, however, can be cold rolled into the product, which is a form of secondary processing that cannot be routinely tested for material properties. Further, cold rolling is a form of strain hardening, which increases hardness in the affected area. This can result in conflicting requirements or industry specification interpretation differences of requirements, which can be problematic with respect to specification compliance.

Due to the general nature of hardness and microstructure impact on susceptibility of materials to hydrogen charging environments, additional hydrogen embrittlement incremental step load testing (i.e. a fracture mechanics-based approach) was also performed. Novel specimen geometries were used for accurate assessment of both machined and cold rolled threads, and the results compared with bulk material properties.

The PHNAs studied in this program were alloy 718–120k (UNS N07718), alloy 718–150k (UNS N07718), alloy 725–120k (UNS N07725), and alloy 945–120k (UNS N09945). All materials tested were manufactured in compliance with API 6ACRA, 1st Edition.

A Study of the Effect of Thread Forming on the Susceptibility of Precipitation Hardened Ni-Based Alloy Fasteners to Hydrogen Embrittlement

1 Scope

This report quantifies the hardness profile of both machined and rolled threads in precipitation hardened nickel-based alloys (PHNAs); studies the effects of both machined and rolled threads on hydrogen induced stress cracking (HISC) susceptibility in relevant PHNAs; tests these PHNA grades used for bolting per the requirements of API standards; and provides guidance for the use of the selected testing methodology and resultant test data in the petroleum and natural gas industry.

2 Normative References

There are no referenced documents that are indispensable for the application of this document.

3 Terms, Definitions, and Abbreviations

3.1 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

3.1.1

damage tolerance index

DTI

A calculated parameter that uses fracture mechanics and mechanical properties to assess the critical defect size under a given stress intensity for a given material.

3.1.2

hydrogen susceptibility ratio

Hsr

A calculated parameter that uses applied stresses and mechanical properties to assess the magnitude of effect a specific thread or surface geometry has on the hydrogen embrittlement susceptibility of a given material.

3.1.3

K_{Ic}

The critical stress intensity under Mode I loading conditions at which the onset of crack growth begins.

NOTE See ASTM E399 for additional information.

3.2 Abbreviations

For the purposes of this document, the following abbreviations apply.

CP	cathodic protection
CT	cut threads
CTOD	crack tip opening displacement
CVN	Charpy V-notched
DTI	damage tolerance Index
DTI_p	damage tolerance index of a given specimen with a threaded geometry (i.e. root radius) exposed to a given environment
EDM	electric discharge machining