



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم

بسم الله الرحمن الرحيم



HANAA ALY



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم



شبكة المعلومات الجامعية التوثيق الإلكتروني والميكروفيلم



HANAA ALY



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم

جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها
علي هذه الأقراص المدمجة قد أعدت دون أية تغيرات



يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



HANAA ALY



Cairo University

STUDY OF THERMO-MECHANICAL TREATMENT AND MN, TI ADDITIONS OF CU-AL-NI SHAPE MEMORY ALLOYS

By

Khaled Ahmed Amin Abdelghafar

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
in
Metallurgical Engineering

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2021

STUDY OF THERMO-MECHANICAL TREATMENT AND MN, TI ADDITIONS OF CU-AL-NI SHAPE MEMORY ALLOYS

By
Khaled Ahmed Amin Abdelghafar

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
in
Metallurgical Engineering

Under the Supervision of

**Prof. Dr. Abdel-Hamid Ahmed
Hussein**

**Prof. Dr. Elsayed Mahmoud
Elbanna**

Professor of Metallurgy
Mining, Petroleum, and Metallurgical
Department
Faculty of Engineering, Cairo University

Professor of Metallurgy
Mining, Petroleum, and Metallurgical
Department
Faculty of Engineering, Some University

**Prof. Dr. Mohamed Abdelwahab
Waly**

Professor of Metal Casting
Foundry Technology Laboratory
Central Metallurgical for R&D Institute
(CMRDI)

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2021

STUDY OF THERMO-MECHANICAL TREATMENT AND MN, TI ADDITIONS OF CU-AL-NI SHAPE MEMORY ALLOYS

By
Khaled Ahmed Amin Abdelghafar

A Thesis Submitted to the
Faculty of Engineering at Cairo University
in Partial Fulfillment of the
Requirements for the Degree of
MASTER OF SCIENCE
in
Metallurgical Engineering

Approved by the
Examining Committee

Prof. Dr. Abdel-Hamid Ahmed Hussien, Thesis Main Advisor

Prof. Dr. Elsayed Mahmoud Elbanna, Advisor

Prof. Dr. Mohamed Abdelwahab Waly, External Advisor

- Emeritus Professor at Foundry Technology Lab, Central Metallurgical Research and Development Institute (CMRDI)

Prof. Mahmoud Mohamed Ibrahim Tash, Internal Examiner

Prof. Dr. El-Zahraa Mohamed Yehia El-Baradie, External Examiner

- Emeritus Professor at Non-Ferrous Lab, Central Metallurgical Research and Development Institute (CMRDI)

FACULTY OF ENGINEERING, CAIRO UNIVERSITY
GIZA, EGYPT
2021

Engineer's Name: Khaled Ahmed Amin Abdelghafar
Date of Birth: 25/ 10 / 1992
Nationality: Egyptian
E-mail: Khaled.ahmed92@hotmail.com
Phone: 01112765006
Address: 25 Omar Elmokhtar, Samy Elgamal,
Mansoura, Egypt
Registration Date: 1 / 10 / 2017
Awarding Date: / / 2021
Degree: Master of Science
Department: Mining, Petroleum and Metallurgy Engineering



Supervisors:

Prof. Dr. Abdel-Hamid Ahmed Hussein, (Thesis Main Advisor)
Prof. Dr. Elsayed Mahmoud Elbanna, (Advisor)
Prof. Dr. Mohamed Abdelwahab Waly, (Advisor)
Central Metallurgical R&D Institute (CMRDI)

Examiners:

Prof. Dr.: El-Zahraa Mohamed Yehia El-Baradie , (External Examiner)
Central Metallurgical R&D Institute (CMRDI)
Prof. Dr.: Mahmoud mohamed Ibrahim Tash, (Internal Examiner)
Prof. Dr. Abdel-Hamid Ahmed Hussien, (Thesis Main Advisor)
Prof. Dr. Elsayed Mahmoud Elbanna, (Advisor)
Prof. Dr. Mohamed Abdel Wahab Waly, (Advisor)
Central Metallurgical R&D Institute (CMRDI)

Title of Thesis:

STUDY OF THERMO-MECHANICAL TREATMENT AND Mn, Ti ADDITIONS OF
Cu-Al-Ni SHAPE MEMORY ALLOYS

Key Words:

Shape memory alloy; martensitic transformation; austenite; solution treatment; X-Phase.

Summary:

There is no comprehensive study on the combined effect of both thermomechanical treatment and modification with alloying elements on the shape memory properties of Cu-Al-Ni shape memory alloy (SMAs).

Therefore, this work aimed at studying the effect of 85% thickness reduction through hot deformation as well as heat treatment on the martensitic transformation of Cu-Al-Ni-Ti-Mn SMAs. The samples were divided into two groups: the first group was solution treated at 900°C for 30 min. (S.T), and the other group was hot-rolled at 900°C followed by solution treatment at 900°C for 30 min. (H.R.S.T). The results showed that the hardness of the samples decreased with increasing the percent of Ni. The DSC investigations revealed that the martensite start temperature (Ms) decreased due to the addition of both Ti and Mn compared to the predicted Ms reported in the literature.

Disclaimer

I hereby declare that this thesis is my own original work and that no part of it has been submitted for a degree qualification at any other university or institute.

I further declare that I have appropriately acknowledged all sources used and have cited them in the reference section.

Name: Khaled Ahmed Amin Abdelghafar

Date: / /2021.

Signature:

Acknowledgments

I would like to express my deep regards and sincere gratitude to Prof. Dr. Abdel-Hamid A. Hussein, Faculty of Engineering, Cairo University for his care, kind supervision, encouragement, constant efforts, and valuable stimulating guidance and fruitful discussion throughout this study.

I offer my profuse thanks with humble reverence to Prof. Mohamed Waly, Foundry Technology Laboratory, Central Metallurgical Research and Development Institute (CMRDI), for his invaluable guidance and support. He was a beacon light, whose constant efforts and encouragement proved to be a parallel stimulus in completing this research successfully.

I would like to thank Prof. Dr. El-Sayed M. El-Banna, Faculty of Engineering, Cairo University for his supervision and support.

I am grateful to Dr. Mervat Ibrahim, Dr. Shimaa El-hadad and Dr. Mohamed Morad, Foundry Technology Laboratory, Central Metallurgical Research and Development Institute (CMRDI), for their support and co-operation in the hours of need and for their expert.

Special thanks to Prof. El-Zahraa Mohamed yahia El-Baradie for giving me the access to her lab facilities.

Special thanks to Prof. Ahmed Ismail Zaky Farahat for giving me the access to metal forming facilities.

Special thanks for Ms. Nadiaa and Mr. Tarek for providing the help in preparing the samples for metallographic investigation.

Last but not least, special thanks are due to the staff of Foundry Technology Laboratory of CMRDI and particularly metallographic, melting, workshop staff for their sincere help.

Table of Contents

DISCLAIMER	I
ACKNOWLEDGMENTS.....	II
TABLE OF CONTENTS	III
LIST OF TABLES.....	V
LIST OF FIGURES.....	VI
NOMENCLATURE	IX
ABSTRACT	X
CHAPTER 1 : INTRODUCTION	1
CHAPTER 2 : LITERATURE REVIEW	3
2.1. INTRODUCTION	2
2.2. PHYSICAL MEANING OF SHAPE MEMORY EFFECT	2
2.3. GENERAL FEATURES OF SMA	5
2.3.1. ONE WAY SME	5
2.3.2. TWO WAY SME	5
2.4. PHENOMENON OF SUPERELASTICTY/PSEUDOELASTICITY	6
2.5. INVESTIGATING TOOLS OF SME.....	7
2.5.1. DIFFERENTIAL SCANNING CALOMETRY ANALYSIS	7
2.6. CATEGORIES OF COMMERCIAL SHAPE MEMORY ALLOYS	9
2.7. COPPER BASED SMAS	9
2.7.1. COPPER ZINC ALUMINUM SMAs	9
2.7.1. COPPER ALUMINUM BERYLLIUM SMAs	10
2.3.1. COPPER ALUMINUM NICKEL SMAs	10
2.8. MANUFACTURING TECHNIQUES OF SHAPE MEMORY ALLOYS...12	12
2.8.1. PROCESSING OF COPPER BASED SMAs	13
2.8.1.1. RAW MATERIALS	13
2.8.1.2. LIQUID METALLURGY ROUTE	13
2.8.1.3. INGOTS FORMING AND SHAPING	13
2.8.1.4. SOLUTION TREATMENT.....	13
2.9. CHARACTERISTICS OF MARTENSITIC TRANSFORMATION	13
2.10. CRYSTALLOGRAPHIC CHARACTERISTICS OF SMAS.....	15
2.11. INFLUENCE OF ALLOYING ELEMENT ADDITIONS ON THE CU- AL-NI SMAS	16
2.11.1. MARTENSITIC START TEMPERATURE OF CU-Al-Ni SMAs	16
2.11.2. INFLUENCE OF Mn & B ADDITIONS ON MECHANICAL PROPERTIES	18
2.11.3. EFFESCT OF BERYLLIUM ADDITION ON MECHANICAL PROPERTIES ..	19
2.11.4. EFFECT OF Ti, Zr, Nb & V ADDITIONS THE MECHANICAL PROPERTIES	19
2.12. APPLICATIONS OF CU-BASED SMAS	20
CHAPTER 3 : EXPERIMENTAL WORK	25
3.1. MATERIALS	25
3.2. SAMPLES PREPARATION	25
3.2.1. CASTING PROCESS.....	25
3.2.2. HOT ROLLING OF SAMPLES.....	26

3.2.3. MACHINING OF ROLLED SHEETS.....	28
3.3. DETERMINATION OF THE TRANSFORMATION TEMPERATURES	30
3.4. HEAT TREATMENT PROCESS.....	31
3.5. MICROSTRUCTURE INVESTIGATIONS.....	31
3.5.1. OPTICAL MICROSCOPE.....	31
3.5.2. SCANNING ELECTRON MICROSCOPE	31
3.6. X-RAY DIFFRACTION ANALYSIS	31
3.5. MICROSTRUCTURE INVESTIGATIONS.....	31
3.7. MECHANICAL TESTS.....	34
3.7.1. HARDNESS TEST.....	34
3.7.2. TENSILE TEST.....	35
3.7.3. COMPRESSION TEST.....	36
 CHAPTER 4 : RESULTS AND DISCUSSION	 37
4.1. MICROSTRUCTURE & XRD ANALYSIS	37
4.2. EDX ANALYSIS.....	41
4.3. RECRYSTALLIZATION OF HOT-ROLLED SAMPLES	44
4.4. TRANSFORMATION TEMPERATURES	46
4.5. MECHANICAL PROPERTIES.....	52
4.5.1. HARDNESS RESULTS	52
4.5.2. COMPRESSION STRENGTH OF S.T SAMPLES	53
4.5.3. TENSILE STRENGTH OF H.R.S.T SAMPLES	53
4.5.4. SHAPE MEMORY PROPERTIES	56
4.6. FRACTURE SURFACE	57
 CHAPTER 5 : CONCLUSIONS	 63
REFERENCES.....	64

List of Tables

Table 2.1: physical properties of commercial shape memory alloys	9
Table 2.2: Categories of shape memory actuators.....	23
Table 2.3: Application window of commercial SMAs.....	24
Table 3.1: Chemical composition of the investigated alloys.....	25
Table 4.1: Summary of chemical analysis of the X-phase precipitates.....	41
Table 4.2: Average grain sizes of the investigated alloys	44
Table 4.3: Thermodynamic data of the martensitic transformation for the S.T samples.	47
Table 4.4: Thermodynamic data of the martensitic transformation for the H.R.S.T samples.	48
Table 4.5: Hardness values of the investigated alloys.....	52
Table 4.6: Summary of mechanical properties for S.T & H.R.S.T samples of the studied alloys.....	55

List of Figures

Figure 2.1: Microscopic demonstration of SME	3
Figure 2.2: Crystallographic changes related to phenomenon of SME.....	4
Figure 2.3: Demonstration of one way SME.....	5
Figure 2.4: Illustration of two-way SME	6
Figure 2.5: Superelasticity behavior	7
Figure 2.6: Standard DSC thermogram for SMA.....	8
Figure 2.7: Strain recovery versus ϵ_{PE}	10
Figure 2.8: 2D section of Cu-Al phase diagram, at 4 % Ni content.....	11
Figure 2.9: Effect of Al percentage on the transformation temperatures (a) M_s & M_f temperatures of entire forward transformation, (b) $M_s(\beta')$, $M_s(\gamma')$, $M_f(\beta')$, and $M_f(\gamma')$, (c) A_s and A_f temperatures of entire reverse transformation, (d) $A_s(\beta')$, $A_s(\gamma')$, $A_f(\beta')$, and $A_f(\gamma')$	12
Figure 2.10: (a) the lattice structure of DO3 austenite, (b) lattice correspondence between 18R martensite and DO3 austenite.....	14
Figure 2.11: Schematic representation of the austenite/martensite interface, identifying the habit plane.....	15
Figure 2.12: (a) DSC thermograms of Cu-Al-Ni SMAs, (b) M_s transformation temperatures as a function of aluminum concentration, and (c) the enthalpy related to Al content	17
Figure 2.13: DSC curves for: (a) base Cu-Al-Ni SMA; (b) modified with 0.2Ti; (c) modified with 0.4Mn; and (d) modified with 0.2Zr	18
Figure 2.14: Stress-strain curves of Cu-based SMA modified with Be at room temperature.....	19
Figure 2.15: Operating and the transformation temperatures for commercial SMAs....	20
Figure 2.16: Application of SMAs micro-actuators in automotive valves.....	21
Figure 2.17: Applications of SMAs in civil engineering (a) isolation system for buildings, (b) spring isolation device, (c) tendon isolation system	22
Figure 3.1: Electric resistance furnace using in melting process	25
Figure 3.2: Pilot scale rolling mill machine	26
Figure 3.3: Macrograph of as-cast ingot (a), hot rolled sample (b).....	27
Figure 3.4: Schematic representation of the used thermo-mechanical cycle	27
Figure 3.5: Macrograph of machined tensile test samples	28
Figure 3.6: (a) compression test samples, (b) as-cast rod.....	28
Figure 3.7: Macrograph of the machine sheets via wire cutting	29
Figure 3.8: Differential scanning calorimetry apparatus	30
Figure 3.9: The used muffle furnace for the heat treatment process	32
Figure 3.10: Zeiss Axiotech 30 optical microscope	32
Figure 3.11: Illustration of average grain size calculation process	33
Figure 3.12: Scanning electron microscope (SEM)	33
Figure 3.13: Hardness test machine.....	34
Figure 3.14: Shimadzu universal testing machine.....	35
Figure 3.15: Designed shape recovery test for H.R.S.T samples	36
Figure 3.16: Typical shape recovery test for S.T samples	36
Figure 4.1: SEM micrographs of S.T samples of the investigated alloys (a) alloy 2N, (b) alloy 3N & (c) alloy 4N.....	38

Figure 4.2: SEM micrographs of H.R.S.T samples of the investigated alloys (a) alloy 2N, (b) alloy 3N & (c) alloy 4N	39
Figure 4.3: XRD pattern of S.T samples	40
Figure 4.4: XRD pattern of H.R.S.T samples.....	40
Figure 4.5: EDX analysis of the X-phase precipitates of S.T samples (a) 2N alloy, (b) 3N alloy, (c) 4N alloy.....	42
Figure 4.6: EDX analysis of the X-phase precipitates of S.T samples (a) 2N alloy, (b) 3N alloy, (c) 4N alloy	43
Figure 4.7: optical micrographs of (a) H.R.S.T sample for alloy 2N, (b) H.R.S.T sample for alloy 3N, (c) H.R.S.T sample for alloy 4N	45
Figure 4.8: heating/cooling DSC curve of S.T samples (a) alloy 2N, (b) alloy 3N & (c) alloy 4N	49
Figure 4.9: Comparison of expected and actual martensite start temperatures.....	50
Figure 4.10: Trend of actual transformation temperatures of S.T samples	50
Figure 4.11: heating/cooling DSC curve of H.R.S.T samples (a) alloy 2N, (b) alloy 3N& (c) alloy 4N.....	51
Figure 4.12: trend of actual transformation temperatures of H.R.S.T samples.....	52
Figure 4.13: Hardness results for the studied alloys.....	53
Figure 4.14: Compression stress-strain curve for S.T samples	54
Figure 4.15: Tensile stress-strain diagram of H.R.S.T samples	55
Figure 4.16: Shape memory recovery ratio of both H.R.S.T & S.T samples.....	57
Figure 4.17: Shape memory effect measurement of S.T samples (a) 2N alloy, (b) 3N alloy, (c) 4N alloy.....	58
Figure 4.18: Shape memory effect measurements of H.R.S.T samples (a) 2N alloy, (b) 3N alloy, (c) 4N alloy	59
Figure 4.19: fracture morphology of polycrystalline Cu–Al–Ni–Mn–Ti alloys (a) S.T sample of 2N alloy, (b) H.R.S.T sample of 2N alloy, (c) S.T sample of 3N alloy, (d) H.R.S.T sample of 3N alloy, (e) S.T sample of 4N alloy & (f) H.R.S.T sample of 4N alloy	60
Figure 4.20: Transgranular cracks of S.T samples (a) 2N ally, (b) 3N alloy, (c) 4N alloy	61
Figure 4.21: Fracture dimples of H.R.S.T samples surrounded by X-phase precipitates (a) 2N alloy, (b) 3N alloy, (c) 4N alloy	62

Nomenclature

SEM	Scanning Electron Microscopy
OM	Optical Metallography
EDS	Energy Dispersive X-Ray Spectroscopy
DSC	Differential Scanning Calorimetry
SMA	Shape Memory Alloy
SME	Shape Memory Effect
18R	Monoclinic Martensite
2H	Hexagonal Martensite
B2	Ordered Body Center Cubic.
L2 ₁	F.C.C superlattice structure
DO ₃	Ordered Austenite
A _s	Austenite Start Temperature
A _f	Austenite finish Temperature
M _s	Martensite Start Temperature
M _f	Martensite finish Temperature
VHN	Vickers Hardness Number