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	HEAT TREATMENT VALIDATION	QA

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HEAT TREATMENTVALIDATION PROCESS

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01		All previous versions of this document are incorporated to develop new issue.	10-09-2024

	Name	Signature	Date
Prepared by:	Arshad Malik QA & RA manager		10-09-2024
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INTRODUCTION

Vacuum Heat Treatment Furnace

SKIVAC vertical vacuum heat treatment furnace; cylindrical shaped with a fixed dished lower end. Constructed of all welded heavy gauge carbon steel plate pressure vessel to BS5500 specifications, the main body is surrounded by a steel cooling jacket which is welded to the body. Into the body is fitted a cooling water coil which immediately surrounds a further cylinder, made of graphite and stainless steel.

This graphite casing acts as insulation jacket for the graphite heating elements, which is turn surround the work pieces, these being carried from a centrally mounted shaft supported top and bottom. The furnace tank and its load are vertically mounted having a removable top, on this top is affixed an electric motor which drives a centrifugal fan. Ports are provided where required for both incoming and outgoing connections.

The control of the furnace is fully automatic; any variation of the heat treatment being easily set into the micro-processer based program and memory system. In addition, a manual override system is provided.

Control Panel

A floor mounted cabinet with front access doors houses the following equipment; Indicating automatic temperature micro-processor controller. Range 0 to 1400°C, complete with Duplex Platinum/Platinum 13% Rhodium thermocouple and compensating cable, capable of giving Ramp 1, Dwell 1, and Temperature 1; Ramp 2, Dwell 2 and Temperature 2; etc. (i.e. multi-segment capability) and end feature, to automatically give slow, cool or rapid quench.

Non-indicating auto excess temperature controller, to a pre-set range of 0 to 1400°C, complete with Plt. /Plt. 13% Rhod. Thermocouple and compensating cable. This is to police the indicating controller and give automatic cut off in the event of an excessive temperature rise. Incorporated in the above is a signal flasher and alarm sound.

Low temperature controller: This is to switch the plant off after the full heating and cooling sequence has been completed, as in the event of night use, or unattended operation. Three phase thyristor unit with integral cooling fan; designed to control the Delta connected load. Triple pole main contractors and thermal overload for heaters, fan, vacuum pump, 91 l/m water pump and 41 l/m water pump.

Triple pole main isolator; being mechanically interlocked with the cabinet door.
Full manual override facility for all functions.

Process Validation Protocol

Title: Heat Treatment Process Validation

Products to be covered: All types of Surgical, Dental & Veterinary Instruments

Equipment/Process to be validated: SKIVAC vertical Vacuum Heat Treatment Furnace/Process and Tempering Furnace

Objective:

Objective of this document is to validate the heat treatment process that consists of Hardening followed by tempering. Vertical vacuum heat treatment furnace and tempering furnace that are made by SKIVAC to improve process flow. The vacuum and tempering furnaces will be validated to assure it performs with existing surgical instruments and existing work procedures. On average 2000 pieces are heat treated per day depending upon size of the instrument, experiments assume this average. The target of heat treatment furnaces is to obtain the hardened and tempered instruments with perfect required hardness in accordance with ISO 7153-1 and microstructures.

Reference Documents:

1. Furnace Operating procedures / manual
2. Basic Instruction to Operate Furnace, SOP - 45
3. Procedure of Hardness Testing Machine, WI/HT/03
4. Validation Procedure; SOP - 14
5. Quality Management Systems – Process Validation Guidance, GHTF/SG3/N99-10:2004 (Edition 2)"
6. Calibration Procedure: SOP - 19
7. Monitoring & Measurement of Product Procedure; SOP - 20
8. ISO 7153-1 - Surgical instruments — Materials —Part 1: Metals
9. Material Safety Data Sheet (if any)
10. Metallographic Test: CMFT/17/18956, CMFT/17/18964,CMFT/17/18961

Validation Plan:

SKIVAC heat treatment furnaces will be subjected to the Installation Qualification, Operational Qualification and Performance Qualification procedures outlined in the Final Documents "Quality Management Systems – Process Validation Guidance, GHTF/SG3/N99-10:2004 (Edition 2)".

The Installation Qualification will utilize the heat treatment furnace's operating manual to define requirements for electrical, plumbing and its intended use. The heat treatment furnaces have been installed, checked and calibrated in production area (Heat Treatment shop). Particular attention will be paid to the atmosphere (ventilation, emergency exit, PPEs etc.) of room, so that the requirement for integrity of the environment is not compromised. A checklist of requirements will be completed and results approved.

Operational Qualification will be completed in three phases. In the first phase, the heat treatment furnaces will be subjected to an initial operation to observe the initial parameters i.e. vacuum level, heating/cooling time and rate, soaking time, required temperature, quenching gas pressure and water flow. Surgical instruments will be heat treated, but detailed assessments of hardness will not be completed. Data for process time, temperature, vacuum level, quenching rate, water flow will be recorded. Variations in these parameters will be subjected to a screening experiment to establish the possible optimal situations.

In the second phase, the process will be centralized and determine first operation capability. The process will be conducted in the production area and with production personnel. Production personnel will be trained on the use of the heat treatment furnaces. Heat treatment furnace's settings for the heating/cooling time/rate, vacuum level, quenching gas pressure, quenching rate, water flow, set temperatures, tempering temperature and soaking at tempering temperature will be used which were determined to be optimal during the first phase of operational qualification. The sampling will be performed randomly as per the size of the lots i.e. 5% of each lot and the results recorded. The variation will be monitored. The number of trials, samples and evaluations will continue until it is determined that the surgical instruments are heat treated evenly. Optimal heat treatment furnace's settings will be determined for the next phase.

In the third phase of operation qualification will be determine the sensitivity of the process by the variation in parameters i.e. in heating rate/time, required temperature for different items and quenching pressure/rate, tempering temperature and soaking at temperature. The normal production process will be used. Production personnel will be trained on the use of the heat treatment furnaces. Trials will be completed 1) with the optimal settings, 2) with a short dwell and long ramp time, low temperature; and 3) long dwell time and short ramp time, high temperature; 4) Low tempering temp., long soaking; 5) high tempering temp., short soaking. Action levels for adjustment of the optimal heat treatment parameters will be determined.

To ensure the quality of heat treatment of all type of surgical and dental instruments the following tests will also carried out. Results specification is given in Table 4 of ISO 7153-1 and Chapter "Martensitic Structures" of ASM Volume 9.

- Hardness Test
- Metallographic Test

Table 4 — Stainless steel

Reference letter according to Table 1	Material number according to EN 10088-1:2014	Material short-term according to EN 10088-1:2014	Hardness in Rockwell (HRC) ISO 6508-1	Hardness in Vickers HV5/ HV10[a] ISO 6507-1	Steel grade
b	1.4006	X12Cr13	35	360	martensitic
e	1.4016	X6Cr17	N.A.	N.A.	ferritic
a	1.4021	X20Cr13	42 - 50	420 - 530	martensitic
b	1.4024	X15Cr13	40 - 48	400 - 500	martensitic
g	1.4028	X30Cr13	49 - 55	510 - 620	martensitic
c	1.4034	X46Cr13	50 - 58	530 - 675	martensitic
m	1.4037	X65Cr13	57 - 61	650 - 750	martensitic
d	1.4057	X20CrNi16-2	40 - 48	400 - 500	martensitic
e	1.4104	X14CrMoS17	30	310	martensitic
e	1.4105	X6CrMoS17	N.A.	N.A.	ferritic
q	(1.4108)[b]	X30CrMoN15-1	54 - 59	590 - 700	martensitic
p	1.4109	X70CrMo15	55 - 60	610 - 720	martensitic
f	1.4112	X90CrMoV18	52 - 60	545 - 720	martensitic
g	1.4116	X50CrMoV15	50 - 58	530 - 675	martensitic
h	1.4117[b]	X38CrMoV15	50 - 58	530 - 675	martensitic
f	(1.4121)[b]	X22CrMoNiS13-1	48 - 54	500 - 600	martensitic
c	1.4122	X39CrMo17-1	50 - 58	530 - 675	martensitic
f	1.4123	X40CrMoVN16-2	52 - 57	560 - 660	martensitic
—	1.4125	X105CrMo17	54 - 60	590 - 720	martensitic
f	1.4197[c]	X22CrMoNiS13-1	48 - 54	500 - 600	martensitic
i	1.4301	X5CrNi18-10	N.A.	N.A.	austenitic
j	1.4305	X8CrNiS18-9	N.A.	N.A.	austenitic
k	1.4310	X10CrNi18-8	N.A.	N.A.	austenitic
l	1.4401	X5CrNiMo17-12-2	N.A.	N.A.	austenitic
m	1.4441	X2CrNiMo18-15-3	N.A.	N.A.	austenitic
o	1.4542	X5CrNiCuNb16-4	selectable	selectable	precipitation hardening
p	1.4543	X3CrNiTiNb 12-9	selectable	selectable	precipitation hardening

[a] The conversion from Rockwell hardness to Vickers hardness is done according to ISO 18265:2003, Table A.1.
To the calculated HV5/HV10 value 1 HRC (value based on experience for stainless steels) was added.

[b] Not included in EN 10088-1. Where the material number is given in brackets, the material was only included in the steel-iron-list.

[c] Historic material number which is used in different fields of application than 1.4121, but is designated to the same material (as 1.4121). 1.4197 is used for rotating instruments.

Performance qualification will perform after completion of operational qualification. Optimal settings forth heat treatment furnace will be used and the action levels for adjustment of heating/cooling time/rate, required temperature for different items and quenching gas pressure & quenching rate will be used. The sampling plan will be used as per lot size of various items and the results recorded. The process will be consider validated when all the parameters are adjusted as per standard ASTM F899 and SOP – 14 will be used to control the process & documents.

Measurement / Testing Equipment and Calibration:

1. Temperature / Time Controller, calibrated per SOP - 19
2. Vacuum gauge, calibrated per SOP - 19
3. Pressure gauge, calibrated per SOP – 19

Equipment Maintenance:

During validation, the heat treatment furnace will be maintained as per the SKIVAC Operating Manual. Upon completion of the validation, the manual will be updated to include maintenance and calibration of the Heat treatment furnace.

Validation Team OQ Results Approval:

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Shahzad Rashid
(Director)

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Jamashid Sarwar
(Production Engineer)

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Arshad Malik
(QA & RA Manager)

_____
M. Ijaz
(Furnace Operator)

Installation Qualification (IQ) Results

Installation Checklist

Requirements were established from the heat treatment operating manual for hardening and tempering and heat treatment working procedure.

Principal Features (for vacuum furnace)	Source	Status
SKIVAC vertical Vacuum Hardening/quenching Furnace	operating manual	Conforms
Model: SV2001-450600	operating manual	Conforms
Operating temperature range: 500 – 1250 ^{0}C	operating manual	Conforms
Normal Process Vacuum: 0.1 m bar	operating manual	Conforms
Ultimate Vacuum: 0.01 m bar	operating manual	Conforms
Electrical Supply: 415 volts, 3 phases, 50 cycles, 4 wires	operating manual	Conforms
Furnace Rating: 55 KW	operating manual	Conforms
Useful charge details:	operating manual	Conforms
– Diameter: 460 mm		
– Depth: 600 mm		
– Capacity: 50 Kg (Uniformly distributed)		
– Capacity Max.: 100 Kg		
Gas Quench Details:	operating manual	Conforms
– Atmosphere (recommended): Nitrogen (High Purity)		
– Back filling pressure, up to: 6 Bar (1 bar normal)		
Cooling water circulation (minimum)	operating manual	Conforms
– Power lead through 10 liters per min		
– Vessel lid and body 20 liters per min		
– Vessel lid and body during quench 85 liters per min		
– Inlet temperature maximum 25 ^{0}C		
– Inlet temperature minimum 10 ^{0}C		
Approx. weight: 3500 Kg	operating manual	Conforms
Installation Drawings	Drawings	Conforms

Principal Features (for tempering furnace)		
SKIVAC vertical Tempering Furnace	operating manual	Conforms
Model: ARC 550	operating manual	Conforms
Maximum rated temp.: 550 ^{0}C	operating manual	Conforms
Electrical Supply: 380/415 Volts 3 Phase 50/60 cycles	operating manual	Conforms
Furnace Load: 15 KW	operating manual	Conforms
Max. Workload dimensions:	operating manual	Conforms
– Diameter: 460 mm		
– Depth: 600 mm		
Approx. combined plant weight: 600 Kg	operating manual	Conforms
Installation Drawings	Drawings	Conforms

First Start-up

The heat treatment furnaces operated as described in the heat treatment operating manual and as required by working procedure.

Calibration

Temperature controller, vacuum gauge, pressure gauge, water flow switches and measuring devices (if any) on the furnaces were successfully calibrated as per calibration requirements.

Issues / Commentary

No new issues were identified.

The environmental challenge of the air exhaust was met by installing exhaust fans in heat treatment shop. The infrastructure / environment matter was monitored as per SA-ICF, no changes from normal levels were detected.

The vacuum heat treatment furnace installation was successful.

Validation Team OQ Results Approval:

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Shahzad Rashid
(Director)

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Jamashid sarwar
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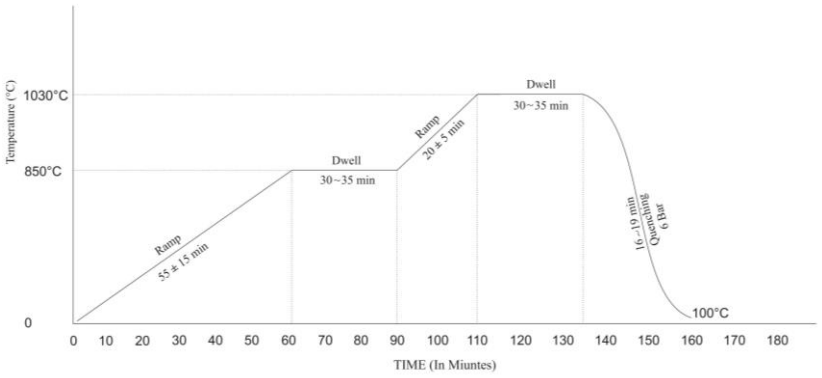
M. Ijaz
(Furnace Operator)

Operational Qualification (OQ) Results

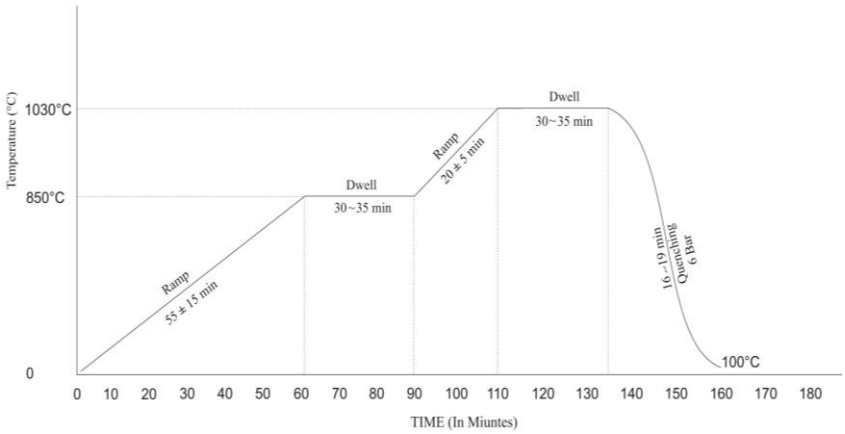
Phase One

Heating rate, heating time, set temperature, soaking time, water circulation (normal), water circulation (during quenching), quenching rate, quenching gas pressure and quenching time were measured with the multiple heat treatment furnace settings for time, temperature and rates. Control graphs of hardening and tempering cycles were completed for multiple items (made up of different material). Control charts were completed as follows:

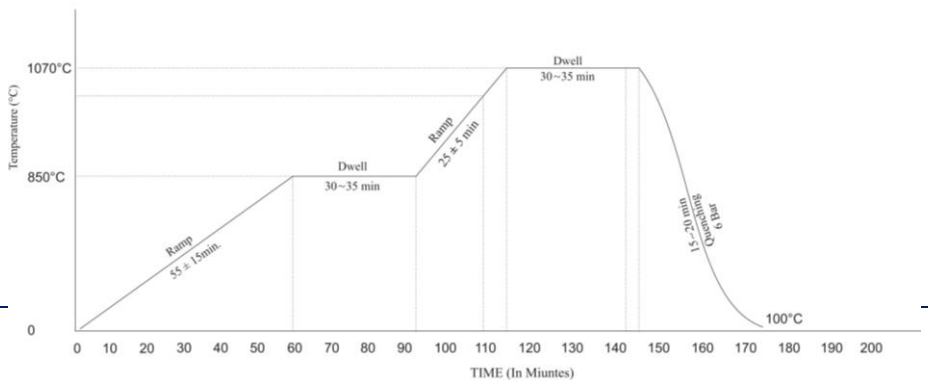
1.4024/ AISI 410

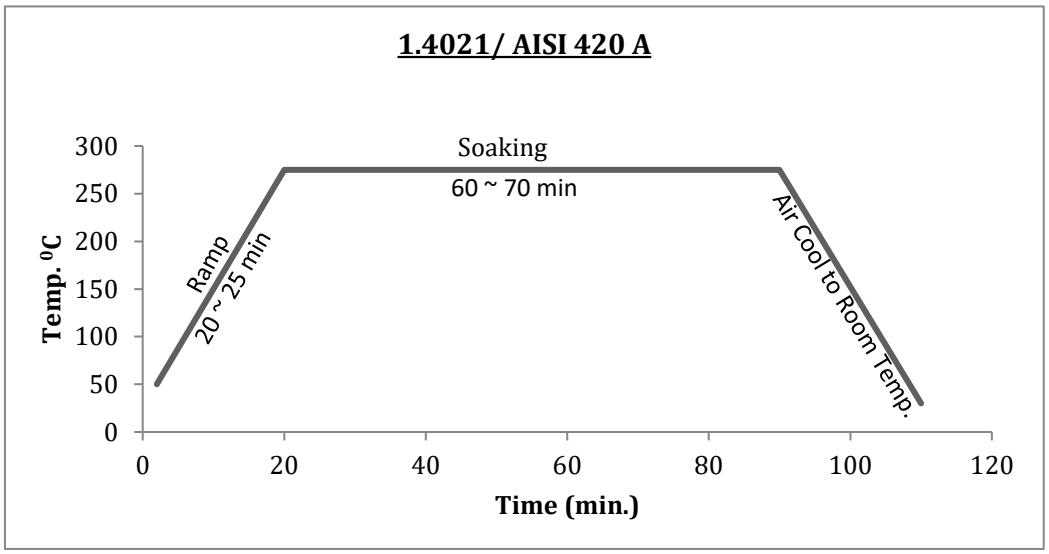


1.4021/ AISI 420 A



1.4028/ AISI 420 B

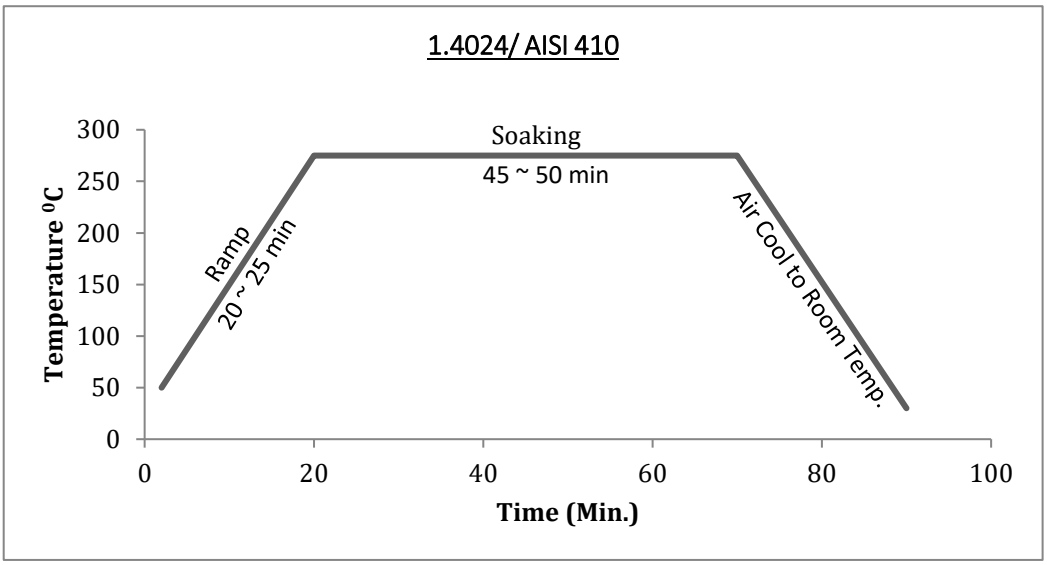


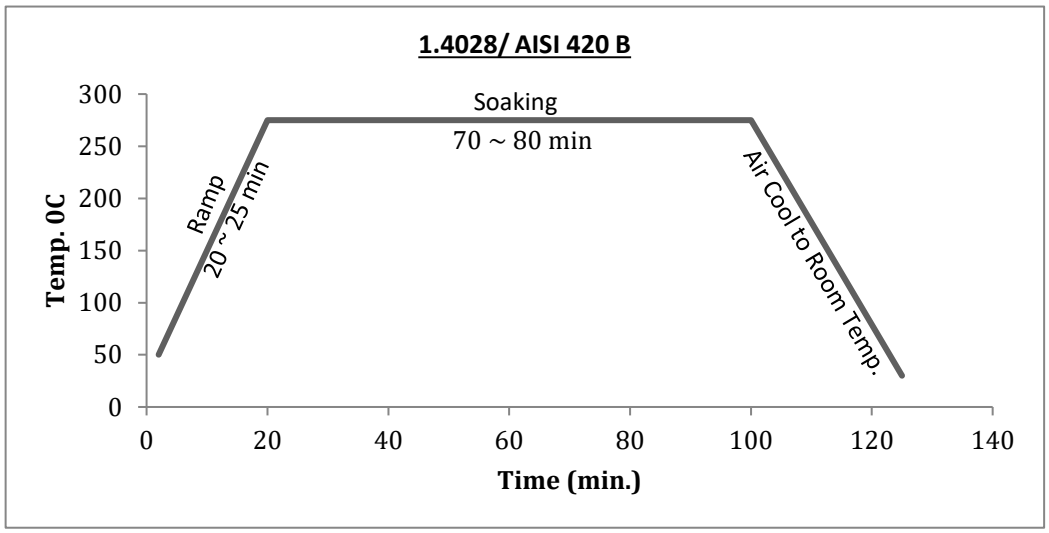


From the analysis of above mentioned graphical representation for different materials, it is apparent that the final set temperature and quenching rate are the most important variables. First optimal heat treatment furnace settings were established: ramp time 60 – 75 minutes, first soaking temperature 850 ^{0}C, first soaking time 25 – 30 minutes, second ramp time 15 – 20 minutes, second soaking time 30 – 35 minutes, second soaking temperature varies as per the requirement of hardness, material type and instrument size from 1020^{0}C to 1070 ^{0}C, quenching time 15 – 25 minutes.

Tempering Cycle

First optimal settings for tempering were also established over a multiple run of furnace and control charts are made for different materials. Control charts were completed as follows;





From above experiments, it is concluded from the above graphs that the time at the set temperature is most important factor. First optimal tempering furnace settings were established: ramp time 20 – 25 minutes, set temperature 275^{0}C, and time at set temperature varies from material to material and hardness requirements from 45 – 80 minutes.

Phase Two

Post Heat treatment studies were conducted to determine the effects of key inputs on hardness and microstructures. Variations in settings were used and the resultant hardness for multiple trials of each family i.e. scissor, forceps& needle holders, tissue & dissecting forceps, bone holding & Extracting forceps and Retractorswere calculated. The following tables summarize the results of heat treatment.

Equipment Number: VF - 01 & VF - 01	**Variable Parameter:** Quenching rate & Tempering time
Date: 15-06-2012	**Production Stage:** Heat Treatment Process

Product: Scissor				**Material Type:** 1.4028/ AISI 420B			
Trial	Product	Lot No.	Qty	Quench Rate ^{0}C/m	Tempering Time (min)	HRc	Result
1	J-22-123	86993-00585-002	300	70	70	50	OK
2	J-22-127	90600-00468-002	400	71	80	51	OK
3	J-22-075	94322-00549-002	150	69	70	51	OK
4	J-22-007	97541-00738-002	300	70	75	52	OK
5	J-22-022	84823-00345-002	600	70	75	52	OK
6	J-22-855 sc	94159-36453-002	450	72	75	51	OK
7	JT-22-810	89758-06405-002	250	70	65	52	OK

Product:Implant Cutter				Material Type: 1.4028/ AISI 420B			
1	JO-21-1048	70783-15072-001	300	70	65	52	OK
Hardening Temp. (Constant): 1070 ^{0}C				*Tempering Temp.(Constant): 275 ^{0}C*			

Product:Forceps / Needle Holders				Material Type: 1.4024/ AISI 410			
Trial	Product	Lot No.	Qty	Quench Rate ^{0}C/m	Tempering Time (min)	HRC	Result
1	J-17-533	80754-00899-002	500	67	60	44	OK
2	J-17-440	98316-00950-0012	200	69	70	45	OK
3	J-17-058	88219-00923-0012	800	67	70	45	OK
4	J-27-055	98634-01691-001	1000	65	65	44	OK
5	J-27-189	968975-0180-001	800	67	65	44	OK
6	JT-21-975	98540-08573-002	350	68	60	44	OK
7	JT-24-616	97550-09087-0012	250	67	60	43	OK
Hardening Temp.(Constant): 1020^{0}C				*Tempering Temp. (Constant): 275 ^{0}C*			

Product:Tissue & Dissecting Forceps				Material Type: 1.4024/ AISI 410			
Trial	Product	Lot No.	Qty	Quench Rate ^{0}C/m	Tempering Time (min)	HRC	Result
1	J-16-016	89735-00713-002	450	67	70	44	OK
2	j-16-029	97475-00722-001	500	65	60	45	OK
3	j-16-0329	87753-00672-002	600	67	75	44	OK
4	j-16-1107	87634-00755-001	1000	67	65	44	OK
5	j-16-099	97021-00781-002	900	66	70	43	OK
Hardening Temp(Constant).: 1020 ^{0}C				*Tempering Temp.(Constant): 275 ^{0}C*			

Product: Retractors				Material Type: 1.4021/ AISI 420			
Trial	Product	Lot No.	Qty	Quench Rate ^{0}C/m	Tempering Time (min)	HRC	Result
1	J-19-330	96455-01555-002	400	67	70	45	OK
2	J-19-320	93876-01445-002	200	67	70	44	OK
3	J-19-350	86972-01312-001	250	66	65	44	OK
4	J-19-270	84963-01432-001	300	68	75	44	OK
5	J-18-029	90792-15101-002	200	67	60	45	OK
HardeningTemp. (Constant): 1020^{0}C				*Tempering Temp. (Constant): 275 ^{0}C*			

Product:Bone Holding / Extracting forceps				Material Type: 1.4021/ AISI 420			
Trial	Product	Lot No.	Qty	Quench Rate ^{0}C/m	Tempering Time (min)	HRC	Result
1	jo-21-1755	88713-09748-001	300	67	45	45	OK
2	jo-21-799	82754-05399-002	400	68	50	46	OK
3	jo-21-7840	78984-41992-002	200	67	50	46	OK
4	jo-21-1688	70851-08509-001	400	67	60	45	OK

5	jo-21-2515	75940-09607-001	200	65	55	46	OK
Hardening Temp. (Constant): 1030 ^0C				*Tempering Temp. (Constant): 275 ^0C*			

From above experiments it was concluded that irrespective of the size, distribution of charge inside the furnace and heating rate, the main factors that affects the heat treatment of surgical instruments are the final temperature and quenching rate. Surgical instruments that are made up of 1.4021/ AISI 420are properly heat treated at the temperature from 1020^{0}C to 1030 ^{0}C with the quenching rate of 67 ^{0}C/min and hardness ranges from 42 HRc to 48HRc; the instruments that are made up of 1.4028/ AISI 420B are properly heat treated at the temperature from 1050 ^{0}C to 1070 ^{0}C with the quenching rate of 70 ^{0}C/min; hardness ranges from 49HRcto 55HRc; and the instruments made up of 1.4024/ AISI 410 heat treated at 1030 ^{0}C with the quenching rate of 65 ^{0}C/min; hardness ranges from 40 HRc to 44HRc.

Phase Three

Normal production processes were used to perform the heat treatment of the surgical instruments 1) optimal levels; 2) low temperature, and short soaking time; 3) high temperature, and long soaking time 4) slow quenching; 5) Rapid quenching. 10 trials of each family were conducted at different combination of parameters.

Results:

This validation test was conducted with electrically heated and operated SKIVAC vacuum Hardening/Quenching furnace& Tempering Furnace and after the successful validation process the tests showed the instruments are properly heat treated and required hardness and microstructures achieved. This test showed that the suitable final temperature for 1.4021/ AISI 420is 1030^{0}C; soaking time 30 minutes and quenching rate is 67 ^{0}C/min; for 1.4028/ AISI 420B suitable final temperature is 1070 ^{0}C, soaking time is 30 minutes and quenching rate is 70 ^{0}C/min; and for the instruments made up of 1.4024/ AISI 410the suitable final temperature is 1030 ^{0}C, soaking time is 30 minutes and quenching rate is 65 ^{0}C/min.

The heat treatment furnace operated with the slow heating rate to avoid any kind of dimensional distortion of the surgical instruments.

Hardness& Metallographic Test:

Starmed Engineering sponsored hardness and Metallographic inspection according to ISO 7153-1&ASM volume 9to ensure the proper hardness and microstructures requirements from laboratory. The tests are as follows:

- Hardness Inspection
- Metallographic Test

As per the tests conducted in-house & out-source, It has been concluded that required hardness achieved and we obtained the ideal microstructures of Marten site (combination of plate and lath marten site) with no retained or acceptable range austenite (that causes the failure of product). Through the hardness inspection & microstructures studies we concluded that the heat treatment has been done properly.

Attachment:

- Final Report – Hardness Report
- Final Report – Metallographic Report

Lab Notebook Reference

Production Engineer's register.

Issues / Commentary

The input which transmits the most variation to the Heat treatment process is final temperature & quenching rate. The target temperature was adjusted to 1030 ^{0}C for 1.4021/ AISI 420, 1020 ^{0}C for 1.4024/ AISI 410, 1070 ^{0}C for 1.4028/ AISI 420B; soaking time 30 minutes; quenching rates for1.4021/ AISI 420, 1.4028/ AISI 420B and 1.4024/ AISI 410 are 67 ^{0}C/min, 70 ^{0}C/min and 65 ^{0}C/min respectively. The heat treatment furnace Operational Qualification was successful.

Validation Team OQ Results Approval:

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Shahzad Rashid
(Director)

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Jamashid Sarwar
(Production Engineer)

_____
Arshad Malik
(QA & RA Manager)

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M. Ijaz
(Furnace Operator)

Performance Qualification (PQ) Results

Normal productions of surgical instrument's codes (see above tables) were run utilizing lots (see above tables). Optimal heat treatment furnace settings were used. A week of production was completed for each code. The sampling plan will be used as per lot size of various items and the results recorded. Following are control tables:

Scissors			Forceps & Needle Holders			Tissue & Dissecting Forceps			Bone Holding & Extracting Forceps			Retractors		
Tmp. (C)	HRc	Rmk	Tmp. (C)	HRc	Rmk	Tmp. (C)	HRc	Rmk	Tmp. (C)	HRc	Rmk	Tmp. (C)	HRc	Rmk
1070	50	Ok	1030	45	Ok	1020	45	Ok	1030	45	Ok	1020	44	Ok
1060	51	Ok	1020	44	Ok	1030	44	Ok	1040	44	Ok	1030	46	Ok
1070	51	Ok	1030	43	Ok	1020	43	Ok	1030	46	Ok	1020	45	Ok

Therefore, no adjustments were made and no root causes identified.

Lab Notebook Reference:
Production Engineer's register

Issues / Commentary
No new issues were identified. The process has demonstrated stability and capability.

All production and QA employees have been trained and the training schedule in QA-03-01 has been updated accordingly.

Validation Team OQ Results Approval:

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Shahzad Rashid
(Director)

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Jamashid sarwar
(Production Engineer)

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Arshad Malik
(QA & RA Manager)

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M. Ijaz
(Furnace Operator)

Final Report

We have reviewed the requirements of the protocol; the IQ, OQ and PQ reports and compared these to the requirements of the reference documents. All requirements have been met and the process is validated.

Validation Team OQ Results Approval:

_____	_____
Shahzad Rashid	Jamasid Sarwari
(Director)	*(Production Engineer)*
_____	_____
Arshad Malik	M. Ijaz
(QA & RA Manager)	*(Furnace Operator)*

Date:10-09-2024