Contract No:

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Materials Characterization Studies on LANA75/85 Materials for Replacement Beds

Kirk L. Shanahan
December 30, 2016
SRNL-STI-2017-00003, Rev. 1

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LIST OF ABBREVIATIONS

SRNL	Savannah River National Laboratory
TF	Tritium Facility
LANAxx	Where xx is a number. Lanthanum-nickel-aluminum alloy with xx being the subscript on the Al element in the alloy chemical formula, i.e. $LaNi_{4.25}Al_{0.75}$ = LANA75 or $LaNi_4Al$ = LANA100
JMC or JMCUSA	Japan Metals & Chemicals or Japan Metals & Chemicals, USA
SRS	Savannah River Site
SRNL	
HPG	Hydrogen Processing Group (part of SRNL, supports Tritium Facility)
ADS	Analytical Development Section (part of SRNL)
HTRL	Hydrogen Technology Research Laboratory (located in Bldg. 999-2W)
SDS	(Material) Safety Data Sheet
H/M	Hydrogen to Metal atomic ratio (composition)
H	Protium
D	Deuterium
ICP- ES	Inductively Coupled Plasma - Emission Spectroscopy
ICP- MS	Inductively Coupled Plasma - Mass Spectrometry
NAA	Neutron Activation Analysis
SEM/ EDX	Scanning Electron Microscopy/ Energy Dispersive X-ray (Analysis)
XRD	X-Ray Diffraction (Powder Pattern)
XRF	X-Ray Fluorescence

Materials Characterization Studies on LANA75/85 Materials for Replacement Beds [U]

Kirk L. Shanahan

December, 2016

Abstract

During FY15 and FY16, a purchase order (PO) was placed with Japan Metals and Chemicals, USA after an open bidding procurement process for 282 kg of $LaNi_{4.25}Al_{0.75}$ and 226 kg. of $LaNi_{4.15}Al_{0.85}$. These materials were to be used in Tritium Facility replacement beds for existing beds that have reached the end of their useful life. As part of the PO, a 100 g. sample of each material was delivered to the SRNL Hydrogen Processing Group for characterization studies as is typically done for all newly acquired hydride materials. The PO actually employed a "trust but verify" approach where JMCUSA was allowed to ship materials it felt met specifications without SRS confirmation, as long as the data used to do so was delivered to SRS as part of the PO documentation package. Subsequent SRNL analysis revealed that the material met all specifications and was of very high quality. This report documents those findings.

Background

In FY14 the Tritium Facility (TF) determined the upcoming need to replace La-Ni-Al alloy (LANA) –based storage beds due to tritium aging effects on the hydride material. Funding for materials procurement was identified and the replacement effort was initiated by requesting the Hydrogen Processing Group (HPG) of Savannah River National Laboratory (SRNL) to identify the technical specifications that would need to be met by any new material procured. After surveying prior technical specifications and consulting with TF and SRNL personnel familiar with material requirements, a new specification was written and issued (M-SPP-H-00531). Following that a purchase order was initiated (PO# 0000233061).

During the material specification and purchase requisition (PR) writing process, multiple requests were made to vendors for Material Safety Data Sheets (SDS) and unit prices for two materials, LANA75 (nominally $LaNi_{4.25}Al_{0.75}$) and LANA85 ($LaNi_{4.15}Al_{0.85}$). However, the vendor did not supply this information until *after* a PO was placed with them (pricing) and the material delivered (SDS). Because of this, the PR used an estimated price and included a statement that the SDS would not be available until later. The original PO specified the procurement of 182 kg. of LANA75 and 126 kg of LANA85. Once the actual unit price had been determined, it was found to be ~25% of the original estimated price, and funds allowed the purchase of 100 kg more of each material. The PO was then modified to reflect the higher quantities. The PO was finally placed on Sept. 30, 2015.

JMC, USA requested clarification from SRS on the order of manufacture, and SRS replied that the LANA75 material should be manufactured and delivered first, followed by the LANA85 material. JMCUSA then coordinated with their parent company, and the materials were manufactured in JMC's Oguni Plant. The LANA75 material was delivered to SRS on April 21, 2016 and the LANA85 on Aug. 17, 2016. Due to administrative issues, the LANA75 was not delivered to SRNL until May 31, 2016 and the

LANA85 was not delivered until October 10, 2016. The 100 g samples were express mailed and received by SRNL on April 15, 2016 (LANA75) and July 26, 2016 (LANA85).

In retrospect, there were several minor paperwork issues that arose involving the delivery of the samples and bulk materials. All were eventually resolved and the bulk materials were moved to a storage location in the TF. For future reference, the sample delivery at HTRL needs to be specified in the PO, as Receiving was expecting delivery at their facility, and a better way to handle the SDS unavailability was needed, as Receiving procedure was to not accept chemicals without a SDS currently entered into the electronic SDS system. Thus the materials were held briefly in N-Area Receiving until these issues were resolved. Also, the PO should have specified bulk material delivery on-site to the TF instead of to SRNL, as that necessitated a second transfer when they were delivered to HTRL and then had to be moved to the TF.

As soon as the 100g samples were received, work was initiated to characterize the materials. A sample was prepared at HTRL for hydrogen and deuterium absorption/desorption (abs/des) studies to characterize each material's performance and to compare with supplier data. Specifically, how well the material conformed to the specification was determined, i.e. plateau pressure at 80 ^{o}C, plateau flatness, and working capacity were derived from ~80 ^{o}C isotherms. As well, thermodynamic characterization (abs/des enthalpy and entropy) with both hydrogen and deuterium isotopes were conducted. JMC also supplied data packages with the samples and bulk materials detailing their analytical studies, and these are included in Appendix I. Another sample of each material was prepared and delivered to ADS for chemical composition determination by ICP-ES and ICP-MS, Cl determination by NAA, and for physical/chemical characterization by XRD and SEM/EDX analysis. All of these results will be presented below, primarily in Appendices II, III, and IV.

Results and Discussion

The technical specifications (Section 3.1 of the specification) place requirements on the materials' behavior towards hydrogen. A summary of the requirements is given in Table 1. (Note that there was a typographical error in the LANA85 material's original plateau pressure specification. This was corrected with a Supplier Deviation Disposition Request (SDDR-13397, 2/17/2016).) Minimum working capacity, plateau flatness, plateau pressure, and total chlorine content are specified. Pressures are specified in atmospheres and capacity in H/M units, where the H/M number is the ratio of H atoms to metal atoms in the sample.

The first three requirements are to be determined from an 80 ^{o}C hydrogen (protium) desorption isotherm. The last requirement (Cl content) was determined at SRNL by neutron activation analysis (NAA). No signal was detected for Cl in the NAA, thus the analytical result (Table 1.) is that the Cl level is less than the detection limit of the technique as applied to that specific sample.

Figure 1a directly compares the SRNL measured isotherms with the digitized version of the JMC-supplied LANA75 isotherm (original JMC data shown in Appendix I). Note that the digitization process may not be perfect, so the second comparison (Fig. 1b) is a 'photoshopped' overlay of the JMC figure and the SRNL results for comparison. The JMC isotherm seems to drift between the SRNL absorption and desorption isotherms. The reason for this is unknown but may be related to how activated the sample was and/or the parameters used for isotherm determination. Figure 2 presents a favorable comparison of the JMC LANA75 material's isotherms to another LANA75 material used in the 2003 TFM&C project. The original 80 ^{o}C D_2 abs/des isotherm reported in 2004 is supplemented by a recent (2014/15) redetermination of the same isotherm on TFM&C LANA75 that had been shelf-stored since 2003. The difference in isotherms reflects sample-to-sample reproducibility, and illustrate that the differences noted between SRNL and JMC isotherms fall well within normal variation.

Figures 3 and 4 present all the experimentally determined isotherms for the LANA75 material. Figure 5 is the LANA75 van't Hoff plot. Given that only two points are used to construct each van't Hoff line, the precision of the results is not maximal, and the values should be considered estimates only. The linear regression equations for the alpha, plateau, and beta regions are shown on the Figure in the key. Figure 6 presents an example mid-point plateau pressure determination plot using the methodology described above. The plateau region equation (shown on the van't Hoff plot) typically encompassed roughly the range needed for the plateau slope evaluation, and the results listed in Table 1 were computed from those linear regression fit equations. Figures 7 and 8 present all the experimentally determined isotherms for the LANA85 material. Figure 9 is the van't Hoff plot.

The purchase specification required the use of the plateau pressure at Q/M=0.35. This assumes an approximate maximum capacity of ~0.7 Q/M. The data however show a greater span than this, which is reflected in the larger working capacities measured vs. the specified values, so while the use of the 0.35 value is not a problem in the specification, it may not represent the true mid-point composition, and use of the true mid-point is preferred for thermodynamic parameter determinations.

Table 1. Product Requirements vs. SRNL Sample Results

	LANA75		**LANA85**	
	Specification	**Found**	**Specification**	**Found**
Plateau Pressure 80 ^{o}C (atm)	0.26-0.61	0.278, 0.282	0.079-0.237	0.154, 0.152
Plateau Slope (ΔlnP/Δ(H/M))	≤1.25	1.09, 0.84	≤1.25	1.14,1.02
(determined between 0.2-0.5 H/M)				
Working Capacity	>0.67	0.74, 0.74	>0.55	0.628, 0.622
(determined between P(atm))	(0.026-6.1)		(0.086-1.32)	
Chloride Content (ppm)	<250	<119	<250	<123

An isotherm was supplied for each material as part of the deliverables, and is included in Appendix I. (These isotherms are also compared directly to SRNL results in the Figures below.) SRNL verified the materials' performance and extended the effort to include two temperatures and isotherms with deuterium as well. From these isotherms the thermodynamic parameters of enthalpy and entropy of decomposition were estimated by using the plateau pressure values from each isotope at two temperatures. The method used to determine the relevant pressures is to determine the composition midpoint between the intersection points of the plateau with straight lines drawn through the alpha and beta phase regions. The natural log of the pressure values at those mid-point compositions is then plotted against the reciprocal temperature (K), and the slope and intercept of that line calculated. The enthalpy and entropy are then computed per the van't Hoff equation,

$$(1/2)\ln(P) = -\Delta H/RT + \Delta S/R \quad (1)$$

Note that this gives the enthalpy and entropy in terms of moles of H atoms. Often, these values are given in terms of moles of molecular H_2 instead, in which case the values are twice that when expressed per mole of H. Table 2 presents these results in absolute values. (By convention, absorption, which is spontaneous, has a negative sign and desorption a positive one on enthalpy.) Diaz, et al [2] have presented thermodynamic parameters for several LANA alloys, in particular for LANA75. They report an enthalpy of formation of -10.6 kcal/mole H_2 and an entropy of formation of -28.2 cal/mole $H_2/^oK$ (note that they incorrectly state the units as kcal/mole/^oK) (-5.3 kcal/mole H and -14.1 cal/mole H/^oK) This substantially agrees with our values determined here, especially when considering that our values are determined from only two temperature points and thus have a higher percentage of error. Interestingly, it seems to be the entropic term that defines the isotope effect.

Table 2. Thermodynamic Parameters for JMC LANA75 and LANA85 (absolute values)

	Absorption		**Desorption**	
	Enthalpy (kcal/mole H)	**Entropy (cal/mole H/K)**	**Enthalpy (kcal/mole H)**	**Entropy (cal/mole H/K)**
LANA75				
Protium	**5.31**	**14.01**	**5.36**	**13.99**
Deuterium	**5.54**	**14.82**	**5.56**	**14.71**
LANA85				
Protium	**5.30**	**13.40**	**5.34**	**13.35**
Deuterium	**5.55**	**14.32**	**5.71**	**14.54**

Figures 10 and 11 show the XRD spectra obtained from the new materials. The spectra indicate high quality materials. There are no spurious peaks detected, and the observed peaks are sharp. Contaminants (either other phases or elements) would lead to broadening and/or peak shoulders. Figure 12 is a difference spectrum computed by subtracting the scaled spectra. Scaling was accomplished by dividing all spectrum data points by the maximum value from the original spectrum (normalization). This did not match intensities in the strongest peak, so there are still strong peaks in the difference spectrum that reflect this. The intensity differences may be dependent upon the distribution of reflective planes presented to the x-ray beam in each specific sample, and thus should not be considered as informative by themselves and thus are not particularly relevant.

What is relevant is the observation of the characteristic structure obtained when subtracting two similar but offset peaks. This shape is illustrated with an artificial case in Figure 13. Note that peak “A” follows the “B” peak in Figure 13. If the reverse were true the characteristic shape observed would also be reversed, i.e. first a positive going peak that then transitions into a negative going one. The typical structure can be seen in several places in Figure 12, which simply indicates that the XRD peaks of LANA85 are slightly offset from LANA75 as expected. This feature can be seen in the difference spectrum most clearly for the negative peaks at ~35.4, 42.1, 58.5, 62.6, 63.76, and 68.2 degrees (2θ values). This was expected but was very difficult to see without the use of the difference spectrum.

SEM/EDX studies were also conducted and the SEM results are shown in Appendix III. EDX reports are included as reported by ADS in Appendix IV. The XRD, SEM, and isotherm data are all consistent with a very high material quality level. Even so, some morphology differences can be noted.

Material chemical composition was determined by Inductively-Coupled Plasma (ICP)-Emission Spectroscopy (ES) and ICP-Mass Spectrometry (MS). The compositions are very close to stoichiometric with possible slight excess La. Actual computed results from the ICP-ES results are $La_{1.019}Ni_{4.176}Al_{0.824}$ (LANA75) and $La_{1.022}Ni_{4.105}Al_{0.895}$ (LANA85) (calculated assuming the Ni+Al sum is at the nominal 5.0 value). Forcing the Ni values to the nominal ones produces these values: $La_{1.037}Ni_{4.25}Al_{0.839}$ (LANA75) and $La_{1.033}Ni_{4.15}Al_{0.904}$ (LANA85). For the LANA85 sample the La and Al signals were measured. After converting results, reported as μg/g, to moles, the ratio of the Al/La obtained was 0.88, while the nominal value for LANA85 is 0.85. JMC results (see Appendix I) give $LaNi_{4.19}Al_{0.74}$ and $LaNi_{4.14}Al_{0.84}$ when the La value is forced to 1.0, which convert to $La_{1.013}Ni_{4.250}Al_{0.750}$ and $La_{1.005}Ni_{4.158}Al_{0.842}$ if the sum of the Ni and Al is forced to 5.0. Both sets indicate a very slight La excess.

These numbers are overall averages, the actual material may have compositional variations through it, which may be seen in SEM and XRD data in some cases. These numbers imply that both materials have a slightly higher than nominal La and Al content, or alternatively, are slightly Ni deficient.

The ICP-MS technique was used primarily to determine what if any contaminants were present. This was also determined from the ICP-ES reports. The ICP-MS results for LANA75 and LANA85 have positive values reported for Co, Sm, and W contaminants, but these values are not significantly above the check standard’s values (which are above the instrument’s detection limit), and are 4-5 orders of magnitude lower than the counts reported for the primary constituents of La, Ni, and Al. As such, if present at all, they will be on the ppm or 10's of ppm levels, and are in fact probably present in the materials used to dissolve and dilute the LANA samples. The ICP-ES of the LANA75 did not show any signals for possible contaminants. The ICP-ES results for the LANA85 material showed low counts for Li and Mg, but again these will be on the ppm or 10's of ppm level, if truly present. More sample determinations are required to confirm this and solidify the actual level if any. However, the materials are of very high purity.

Prior LANA procurements have generally obtained materials with a slight La deficiency. This was *requested* for the new materials but not required. Both materials may be slightly La-rich (up to ~2-3% depending on how it is calculated and allowing for some variation if additional samples are run), but only one sample was analyzed for each material and that number may be subject to revision if more samples are measured. Excess La is thought to be a potential problem in that La is a getter, and may hold tritium in the material, creating a higher T content than desired when disposing of used material. However, there is no indication of free La in the XRD results (Figures 10, 11, and 12), and some variation in the EDX analyses, possibly indicating that there may be some slight compositional variation that alloys all of the La, eliminating the T gettering concern. (However, EDX signals inherently vary such that the technique is not considered to be fully quantitative.) The materials should be entered into a tritium exposure program to assess this more concretely. Further, tritium aging will induce significant changes in the materials' thermodynamics which needs to be quantified.

Conclusions and Recommendations

These studies have shown that all product requirements were met and the materials are of high quality (minimal inclusions, homogeneous). Tritium aging studies are recommended to assess material property changes with age. It should be noted that recent studies [3] have indicated that the amount of cycling a sample undergoes can affect the extent and type of changes observed in isotherms. In addition, recently published tritium aging studies on Pd alloys [4] have reported extreme sensitivity to cycling. Therefore, tritium aging studies should investigate these issues in La-Ni-Al alloys.

Acknowledgements

This work was funded by DOE under contract number DE-AC09-08SR22470.

References

[1] H. H. Van Mal, et al; "Hydrogen absorption in $LaNi_5$ and related compounds: Experimental observations and their explanation"; J. Less Com. Met. **35** (1974) 65

[2] H. Diaz, et al; "Thermodynamic and Structural Properties of $LaNi_{5-y}Al_y$ Compounds and Their Related Hydrides"; Int. J. Hydr. Ener. **4** (1979) 445

[3] Kirk L. Shanahan, Edward A. Stein; **SRNL-STI-2015-00498**, “Effects of Extreme Operating Conditions on LANA Alloys: Year-End Report, FY15 (Rev. 0)”, September, 2015

[4] Kirk L. Shanahan, “Tritium Aging Effects in some Pd - Cr, Ni, and Co Alloys” in Proceedings of the Tritium2016 Conference, April 17-22, 2016, Charleston, SC; Fusion Science and Technology, 2016, to be published (manuscript **SRNL-STI-2015-00533**).

Figure 1a. Comparison of SRNL 80 ^{o}C H2 Desorption Isotherms and JMC results for new LANA75 material

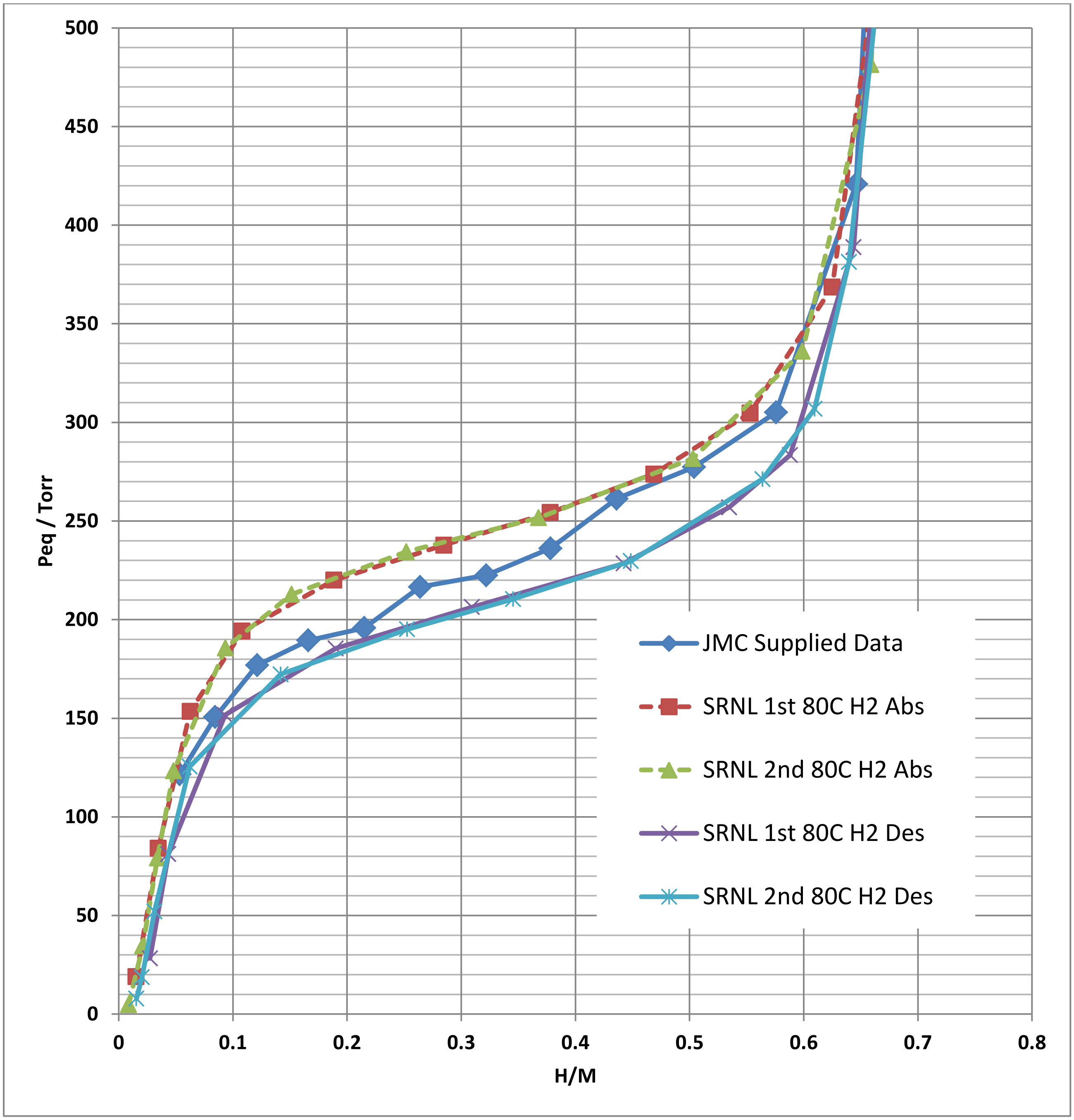


Figure 1b. Photoshop Overlay of SRNL 80 °C H2 Desorption Isotherms and JMC results for new LANA85 material (JMC data is open circles, black line)(SRNL data is closed symbols, colored lines) (P in MPa)

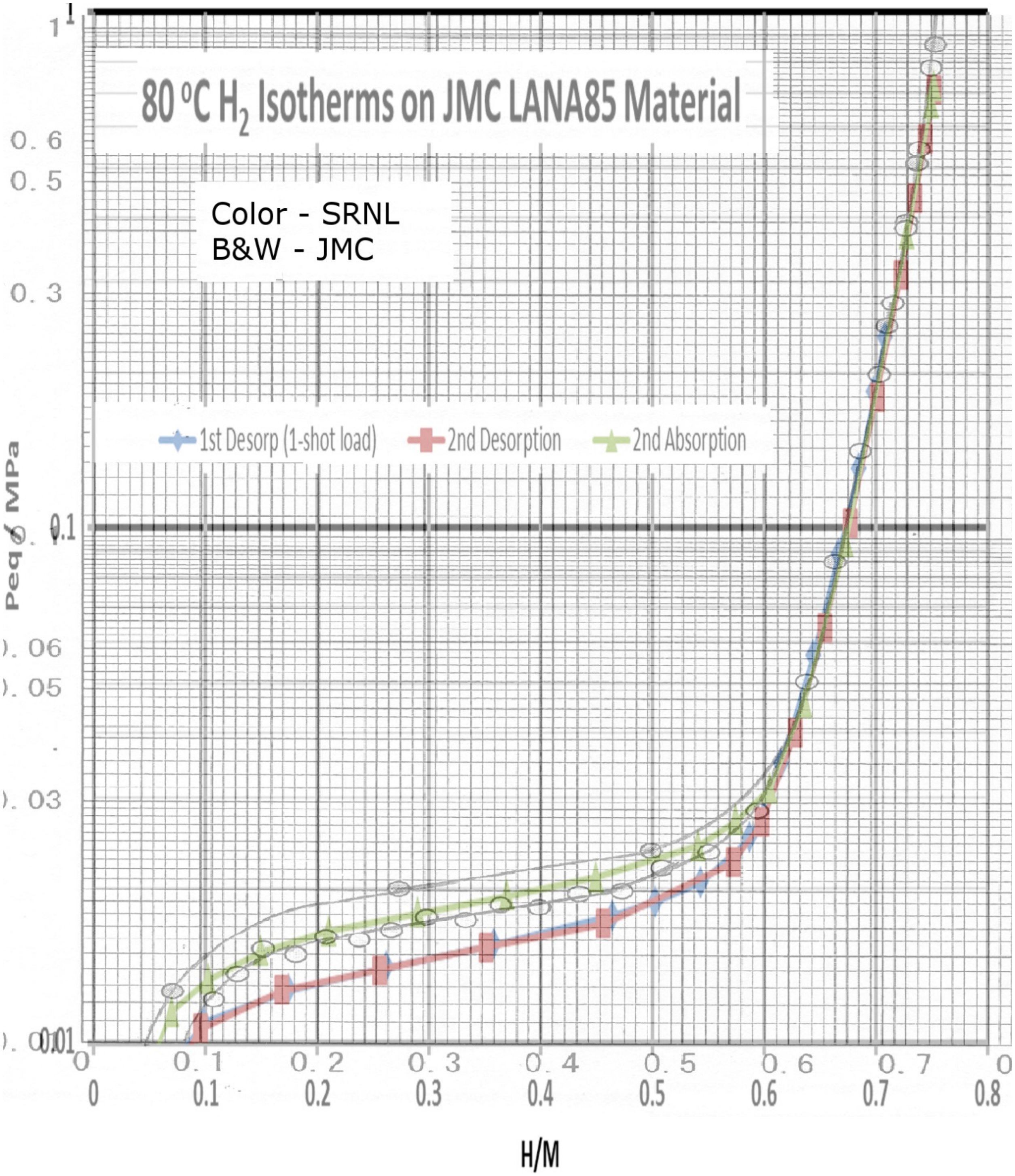


Figure 2. Comparison of new JMC material's 80 °C D_2 isotherms (SRNL data) with two TCON LANA75 batches (Absorptions are dashed lines, desorptions solid)

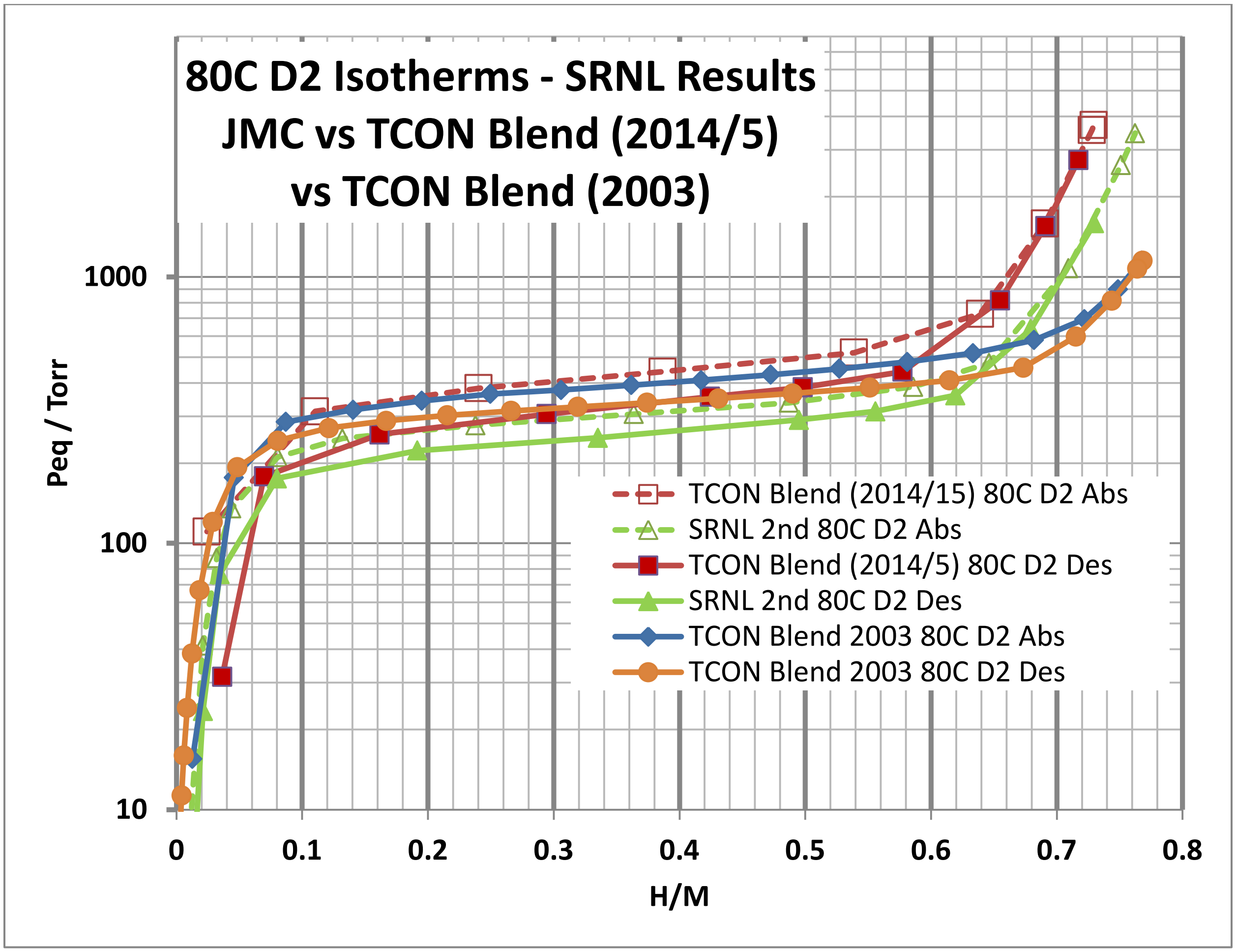


Figure 3. All SRNL data for JMC LANA75. H_2 (red) and D_2 (blue) absorption/desorption (A/D in key) isotherms at 50 & 80 ^{o}C. (Absorption isotherms use dashed lines and unfilled symbols, 1 and 2 designate 1st and 2nd run at that temperature.) (Q= H or D)

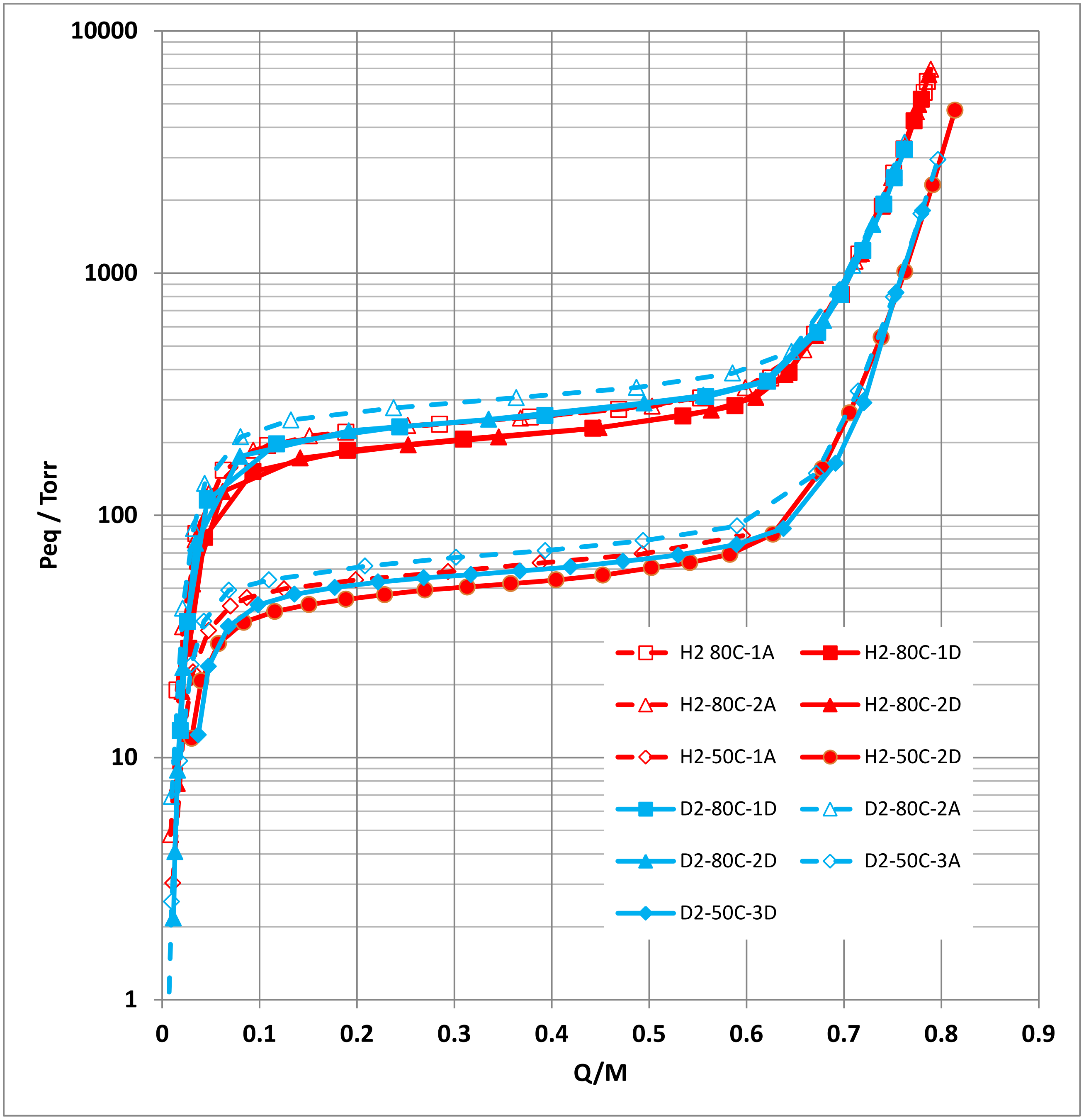


Figure 4. Expanded lower Peq region of Figure 3.

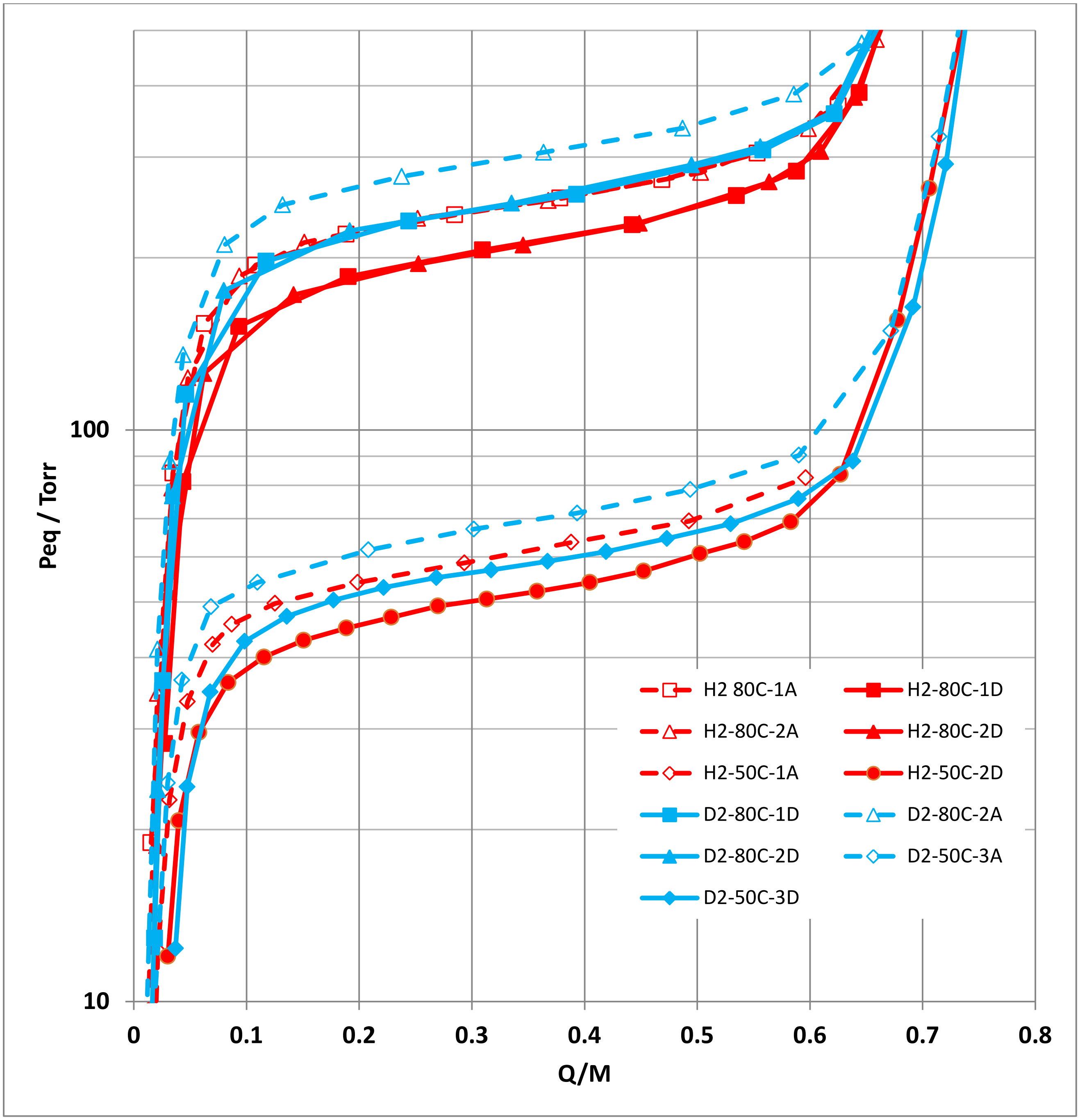


Figure 5. Van't Hoff Plot for JMC LANA75 Material. (Note: Y axis is ½ ln(P_{plat}))

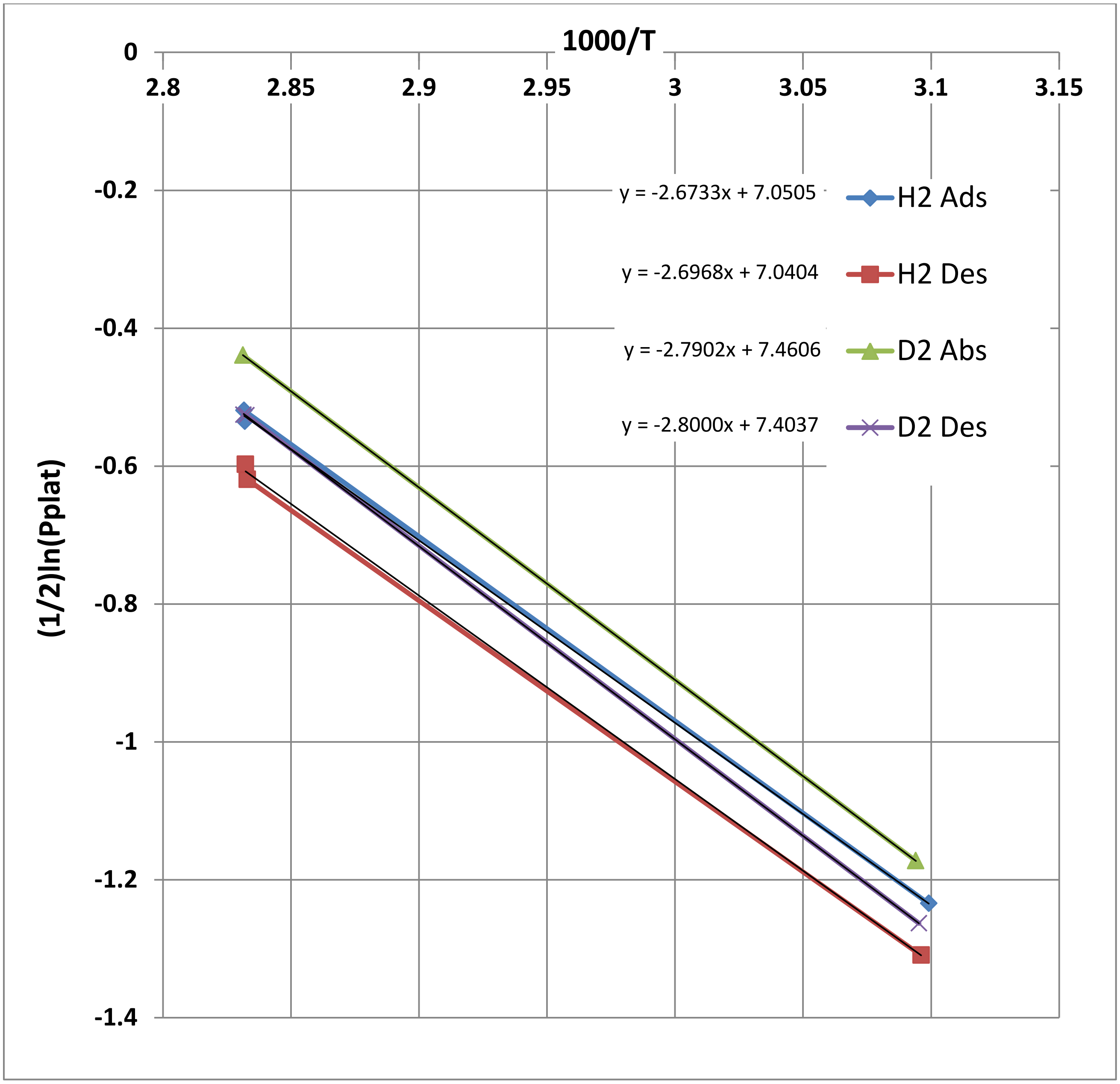


Figure 6. Example Isotherm Analysis for Determining Mid-Plateau Pressure for van't Hoff Plot (linear fits are made to each region of plot, the intercept points define the plateau end points, the mid-point gives P_{plat}). This example uses the 2nd 80 °C D_2 desorption isotherm.

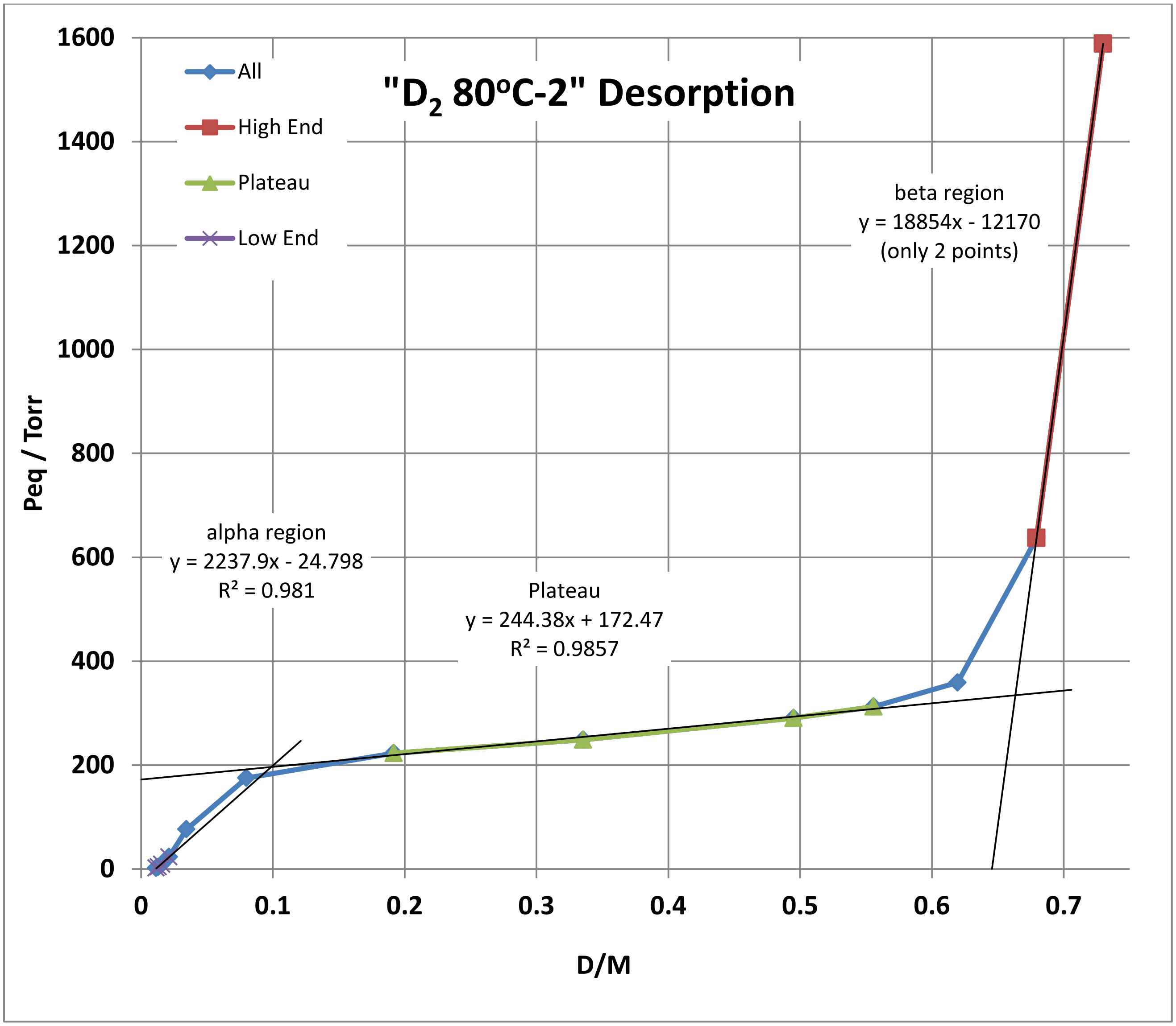


Figure 7. All SRNL data for JMC LANA85. H_2 (red) and D_2 (blue) absorption/desorption (A/D in key) isotherms at 80 & 125 °C (Absorption isotherms use dashed lines and unfilled symbols, 1 and 2 designate 1st and 2nd run at that temperature.) (Q = H or D)

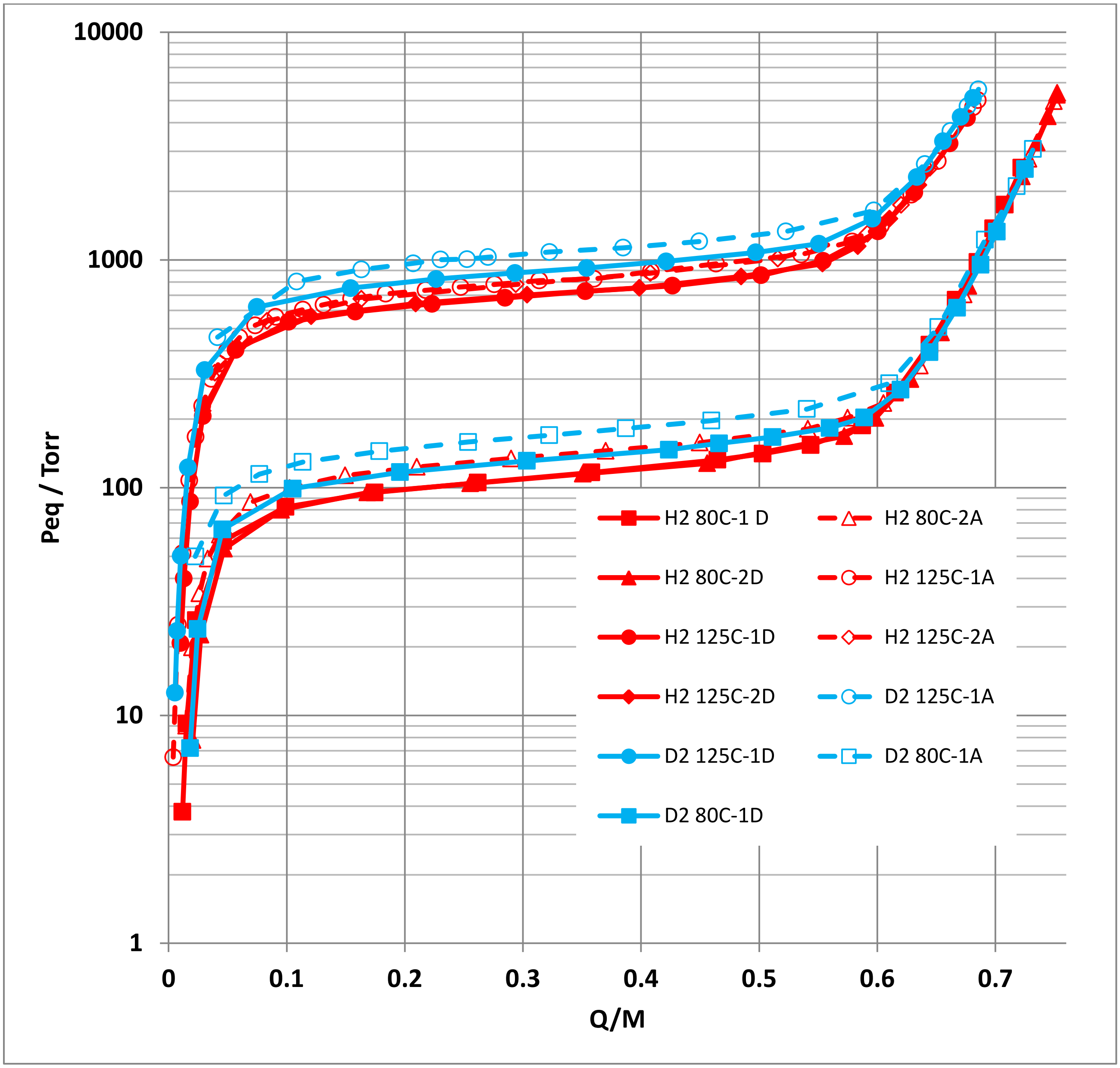


Figure 8. Expanded lower Peq region of Figure 7.

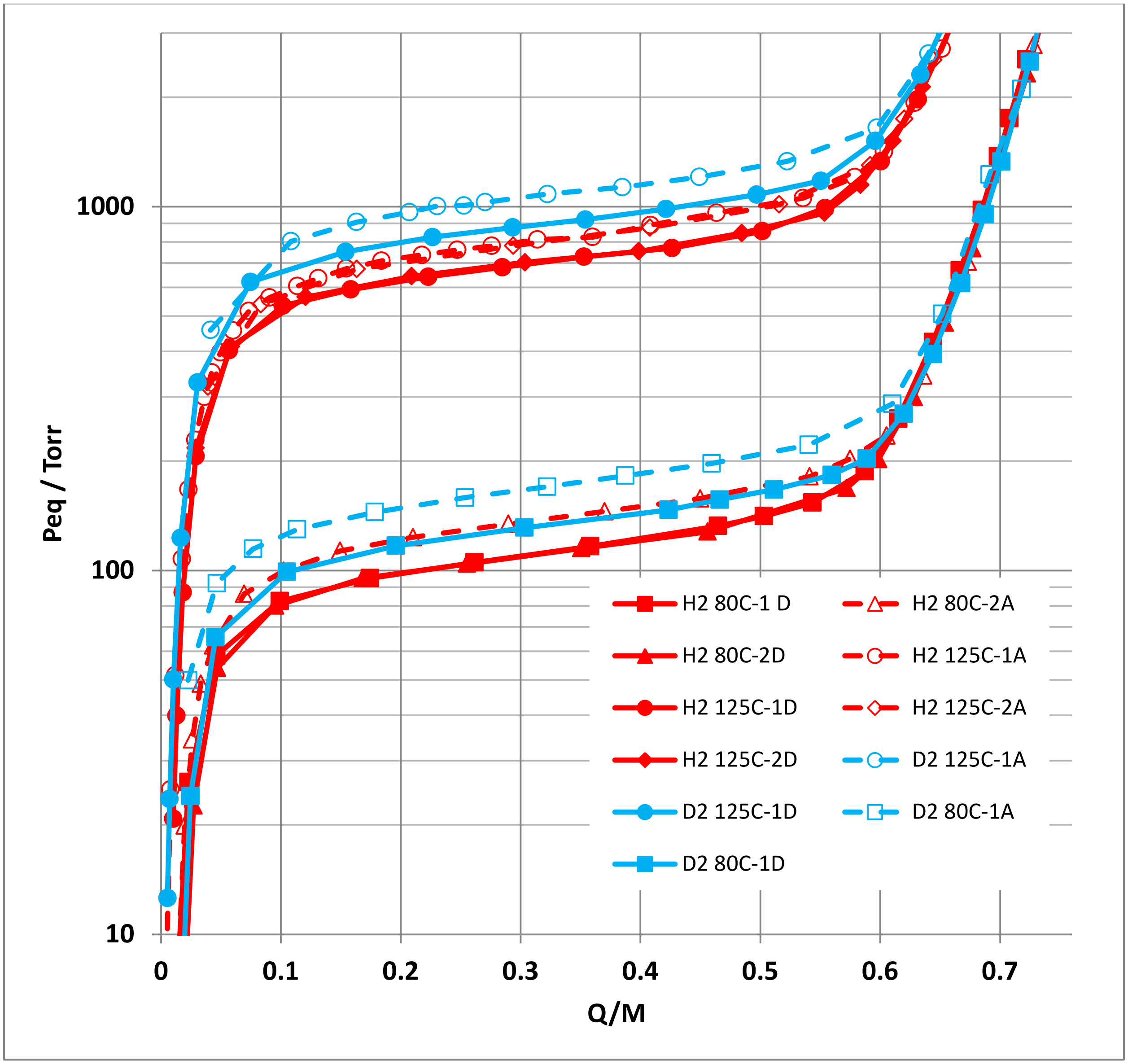


Figure 9. LANA85 van't Hoff Plot (Note: Y axis is ln(P_{plat}), not ½ that)

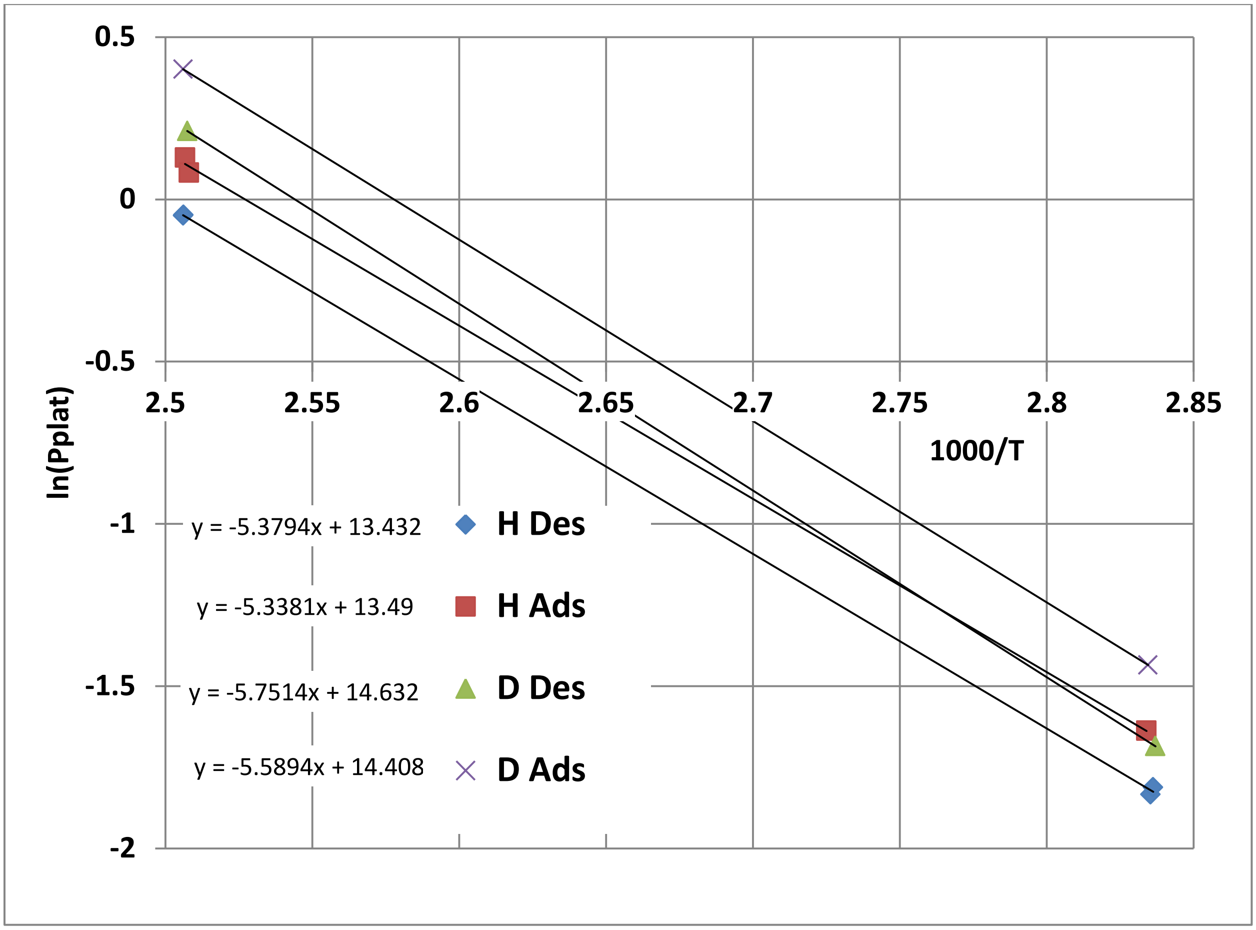


Figure 10. JMC LANA75 XRD Spectrum

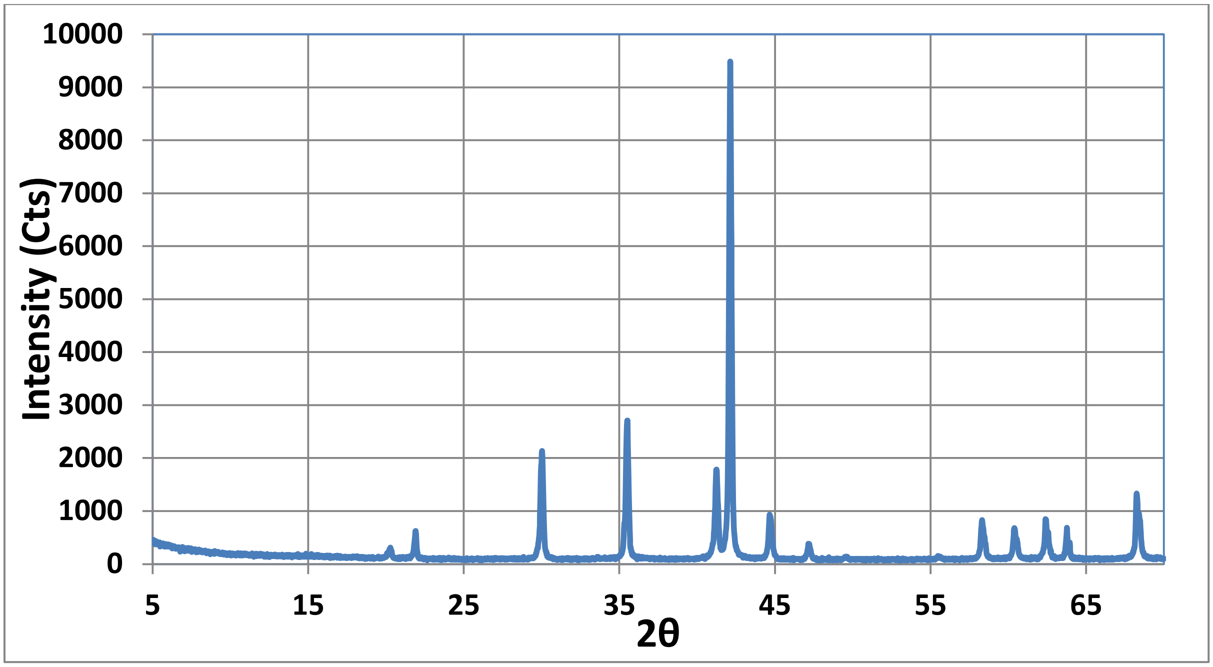


Figure 11. JMC LANA85 XRD Spectrum

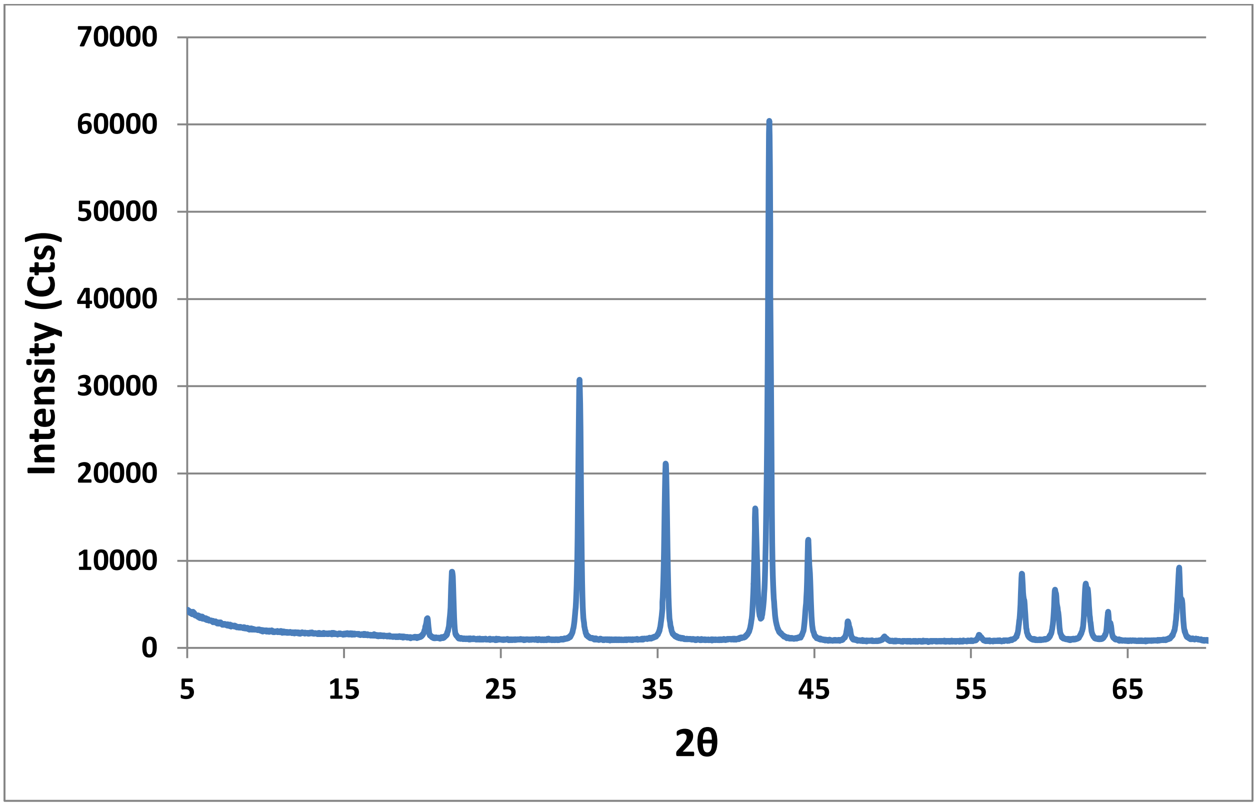


Figure 12. XRD Difference Spectrum (JMC LANA85(scaled)- JMC LANA75(scaled))

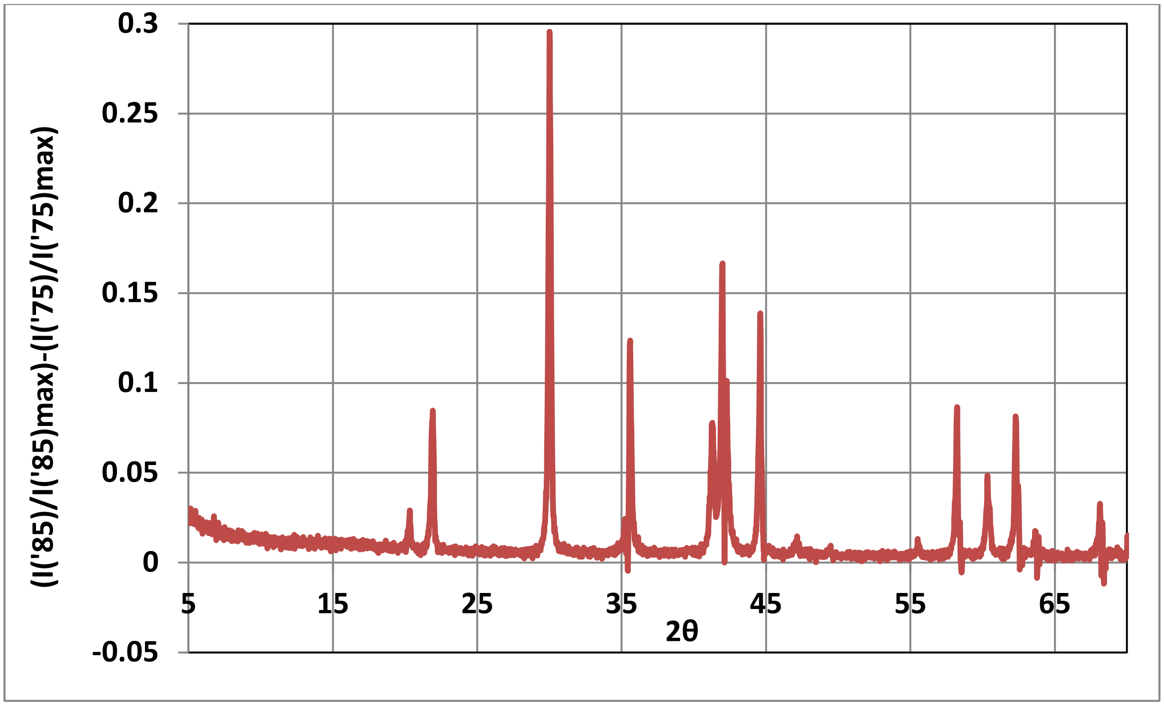
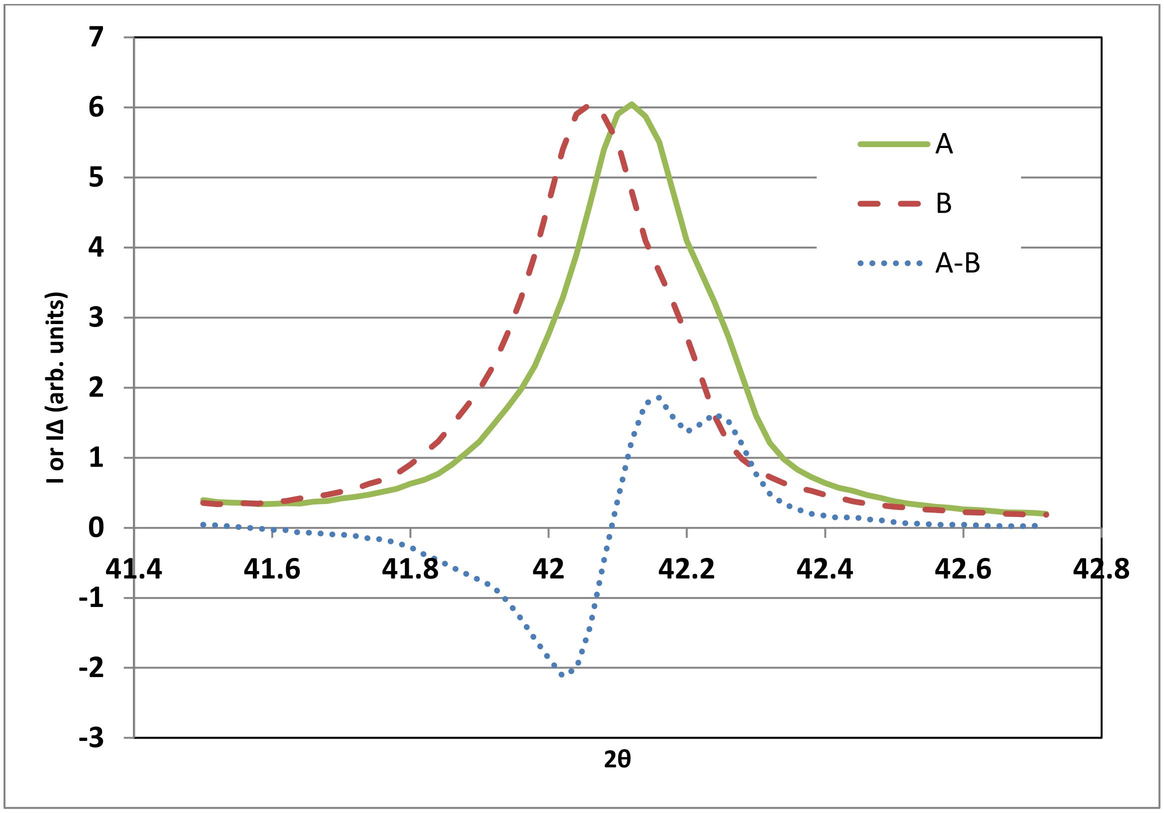


Figure 13. Example of a Difference Spectrum produced from 2 peaks that are slightly offset



Appendix I. JMC supplied data on new LANA materials

Part 1. LANA75

80 °C H_2 Desorption Isotherm

Quality Test Report

Part 2. LANA85

80 °C H_2 Desorption Isotherm

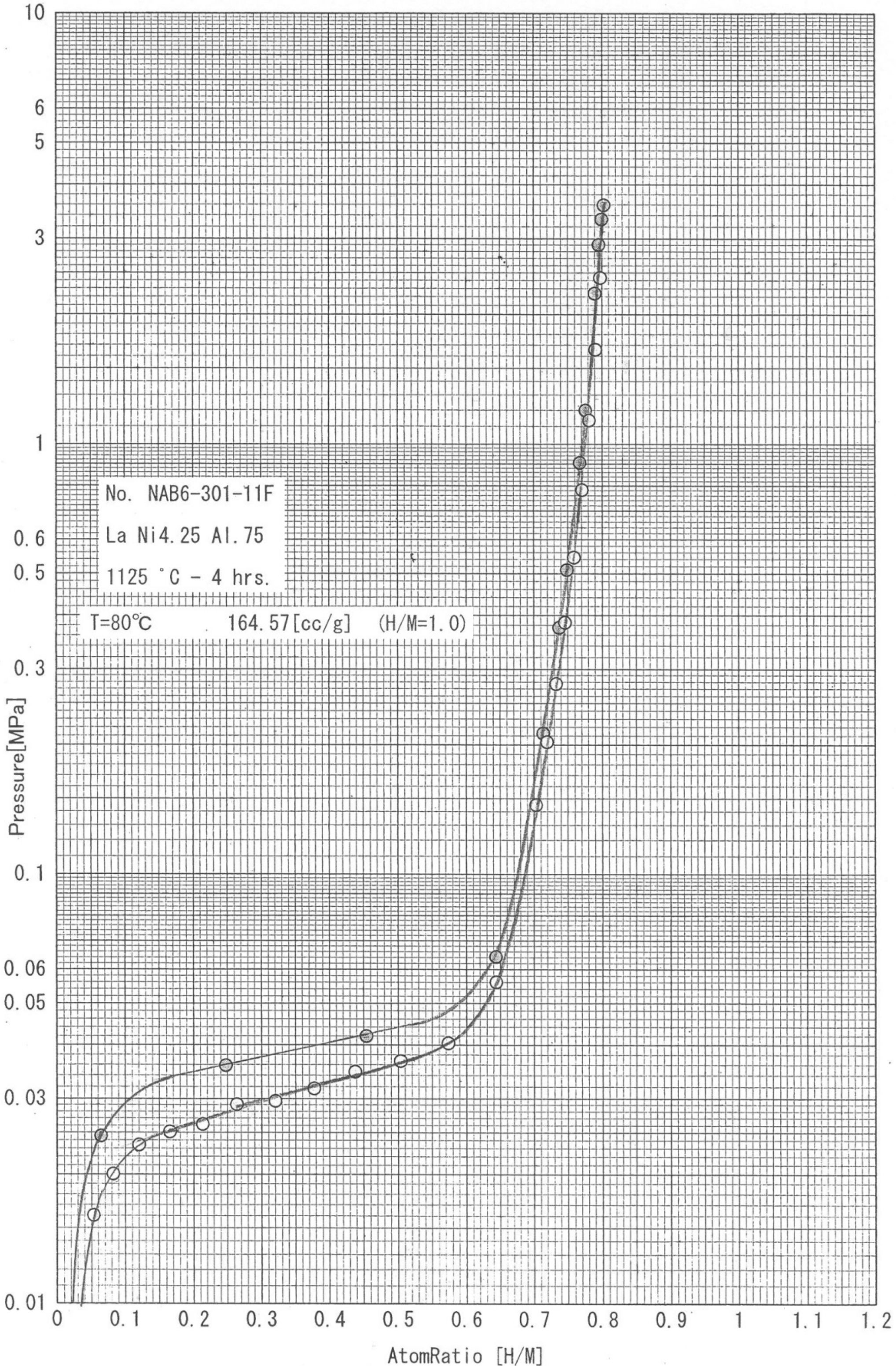
Quality Test Report

Part 3. SDS (applicable to both LANA75 and LANA85)

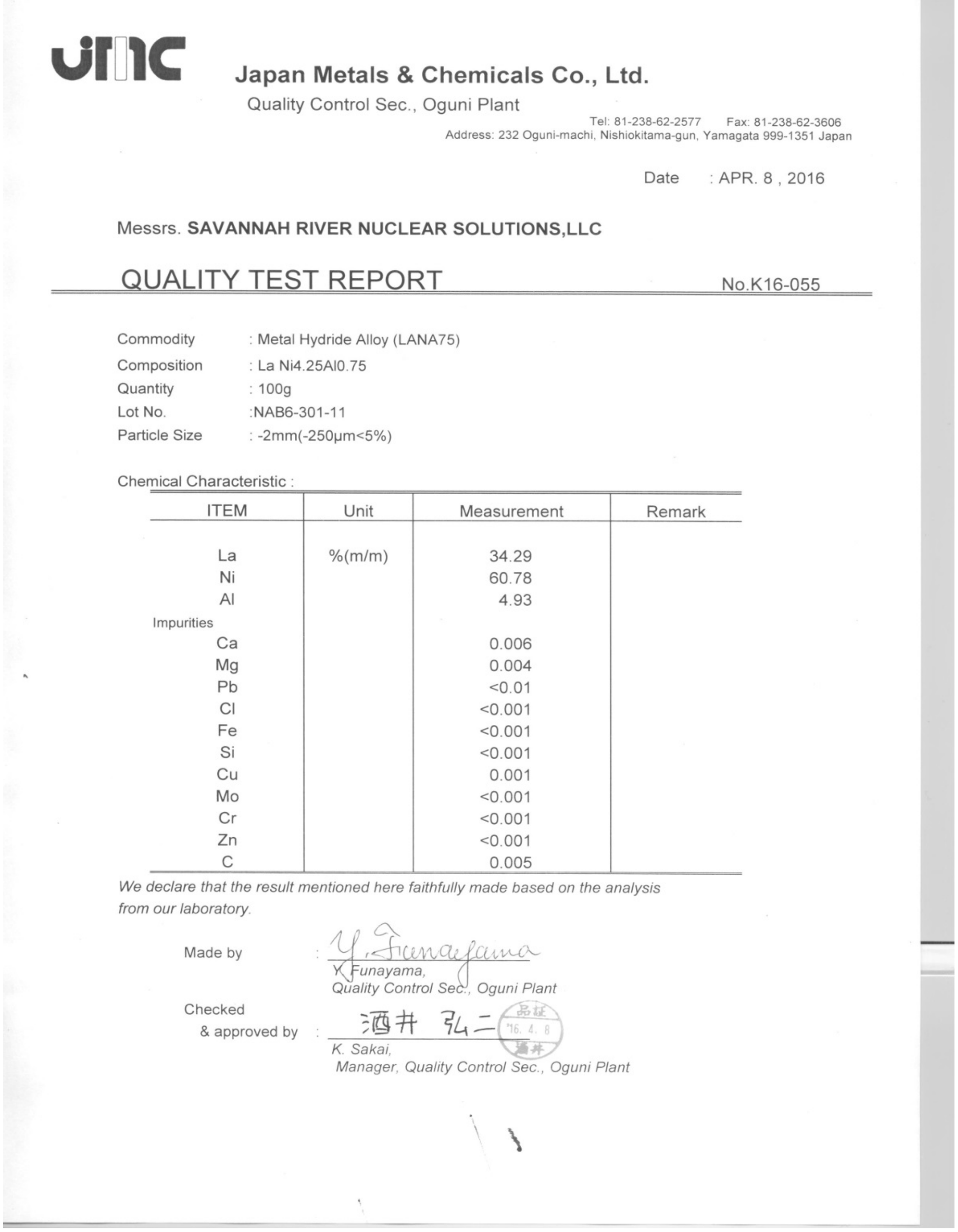
SDS – unactivated material

SDS – activated material

Part 1. LANA75 - 80 °C H_2 Desorption Isotherm



Part 1. LANA75 - Quality Test Report



Japan Metals & Chemicals Co., Ltd.
Quality Control Sec., Oguni Plant
Tel: 81-238-62-2577 Fax: 81-238-62-3606
Address: 232 Oguni-machi, Nishiokitama-gun, Yamagata 999-1351 Japan

Date : APR. 8 , 2016

Messrs. **SAVANNAH RIVER NUCLEAR SOLUTIONS,LLC**

QUALITY TEST REPORT No.K16-055

Commodity : Metal Hydride Alloy (LANA75)
Composition : La Ni4.25Al0.75
Quantity : 100g
Lot No. :NAB6-301-11
Particle Size : -2mm(-250μm<5%)

Chemical Characteristic :

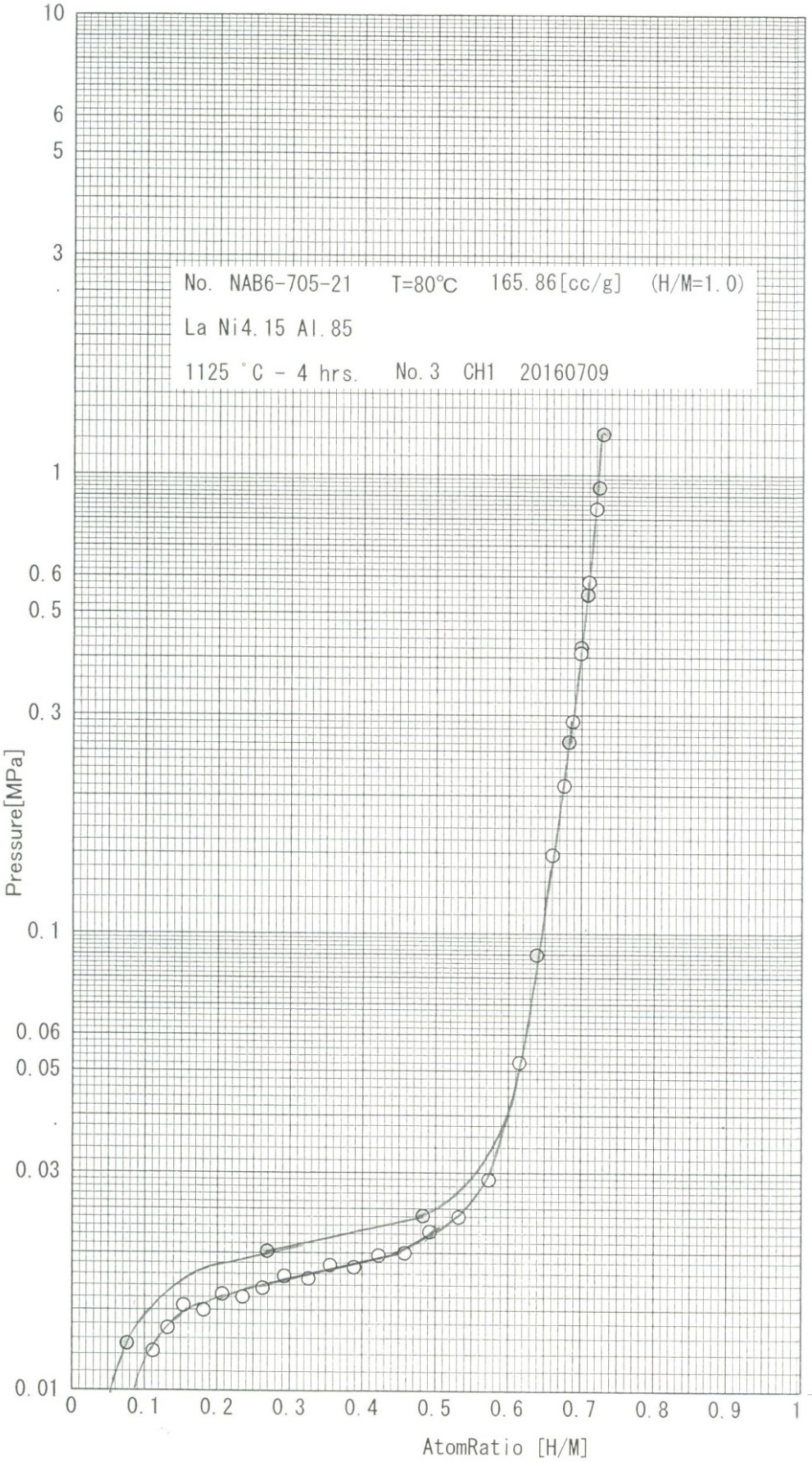
ITEM	Unit	Measurement	Remark
La	%(m/m)	34.29	
Ni		60.78	
Al		4.93	
Impurities			
Ca		0.006	
Mg		0.004	
Pb		<0.01	
Cl		<0.001	
Fe		<0.001	
Si		<0.001	
Cu		0.001	
Mo		<0.001	
Cr		<0.001	
Zn		<0.001	
C		0.005	

We declare that the result mentioned here faithfully made based on the analysis from our laboratory.

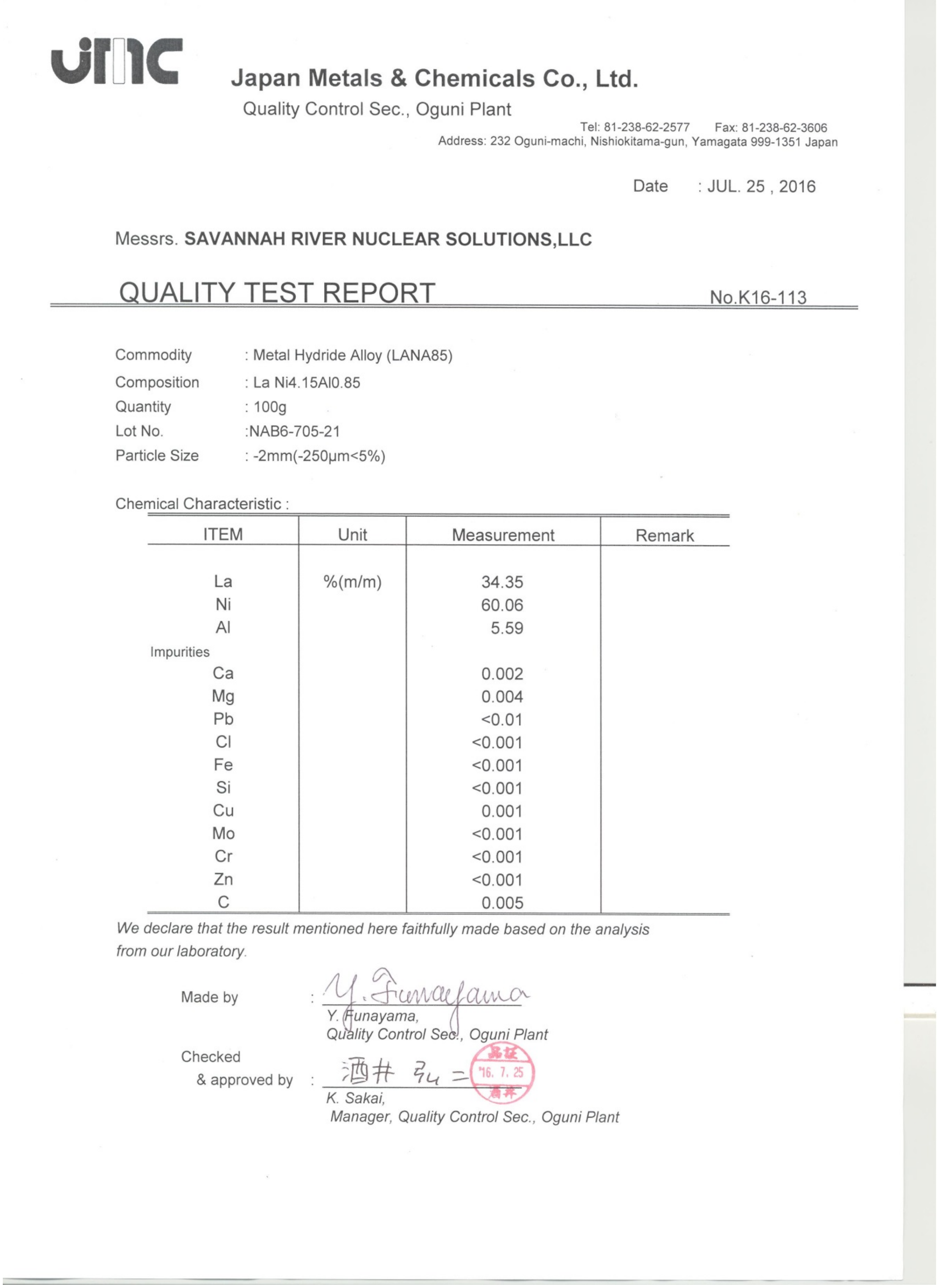
Made by : Y. Funayama
Y. Funayama,
Quality Control Sec., Oguni Plant

Checked
& approved by : 酒井 弘二
品証 '16. 4. 8 酒井
K. Sakai,
Manager, Quality Control Sec., Oguni Plant

Part 2. LANA85 - 80 °C H_2 Desorption Isotherm



Part 2. LANA85 - Quality Test Rep



Japan Metals & Chemicals Co., Ltd.
Quality Control Sec., Oguni Plant
Tel: 81-238-62-2577 Fax: 81-238-62-3606
Address: 232 Oguni-machi, Nishiokitama-gun, Yamagata 999-1351 Japan

Date : JUL. 25 , 2016

Messrs. **SAVANNAH RIVER NUCLEAR SOLUTIONS,LLC**

QUALITY TEST REPORT

No.K16-113

Commodity : Metal Hydride Alloy (LANA85)
Composition : La Ni4.15Al0.85
Quantity : 100g
Lot No. :NAB6-705-21
Particle Size : -2mm(-250μm<5%)

Chemical Characteristic :

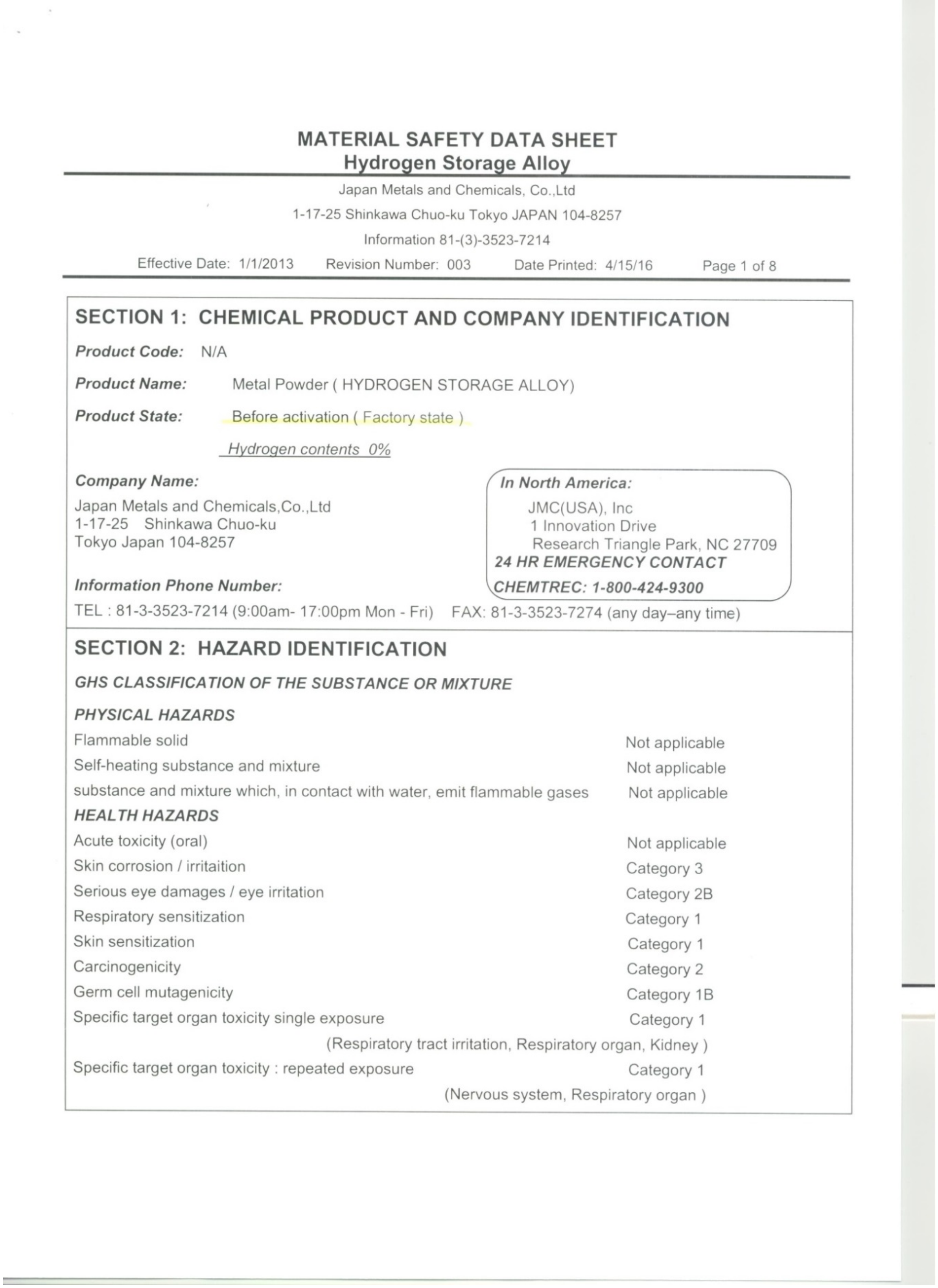
ITEM	Unit	Measurement	Remark
La	%(m/m)	34.35	
Ni		60.06	
Al		5.59	
Impurities			
Ca		0.002	
Mg		0.004	
Pb		<0.01	
Cl		<0.001	
Fe		<0.001	
Si		<0.001	
Cu		0.001	
Mo		<0.001	
Cr		<0.001	
Zn		<0.001	
C		0.005	

We declare that the result mentioned here faithfully made based on the analysis from our laboratory.

Made by : Y. Funayama
Y. Funayama,
Quality Control Sec., Oguni Plant

Checked & approved by : 酒井 弘二
品証 '16. 7. 25 酒井
K. Sakai,
Manager, Quality Control Sec., Oguni Plant

Part 3. SDS – unactivated material (either LANA75 or LANA85)



MATERIAL SAFETY DATA SHEET
Hydrogen Storage Alloy

Japan Metals and Chemicals, Co.,Ltd
1-17-25 Shinkawa Chuo-ku Tokyo JAPAN 104-8257
Information 81-(3)-3523-7214

Effective Date: 1/1/2013 Revision Number: 003 Date Printed: 4/15/16 Page 1 of 8

SECTION 1: CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Code: N/A

Product Name: Metal Powder (HYDROGEN STORAGE ALLOY)

Product State: Before activation (Factory state)

Hydrogen contents 0%

Company Name:

Japan Metals and Chemicals,Co.,Ltd
1-17-25 Shinkawa Chuo-ku
Tokyo Japan 104-8257

In North America:

JMC(USA), Inc
1 Innovation Drive
Research Triangle Park, NC 27709

24 HR EMERGENCY CONTACT
CHEMTREC: 1-800-424-9300

Information Phone Number:

TEL : 81-3-3523-7214 (9:00am- 17:00pm Mon - Fri) FAX: 81-3-3523-7274 (any day–any time)

SECTION 2: HAZARD IDENTIFICATION

GHS CLASSIFICATION OF THE SUBSTANCE OR MIXTURE

PHYSICAL HAZARDS

Flammable solid	Not applicable
Self-heating substance and mixture	Not applicable
substance and mixture which, in contact with water, emit flammable gases	Not applicable

HEALTH HAZARDS

Acute toxicity (oral)	Not applicable
Skin corrosion / irritaition	Category 3
Serious eye damages / eye irritation	Category 2B
Respiratory sensitization	Category 1
Skin sensitization	Category 1
Carcinogenicity	Category 2
Germ cell mutagenicity	Category 1B
Specific target organ toxicity single exposure	Category 1
(Respiratory tract irritation, Respiratory organ, Kidney)	
Specific target organ toxicity : repeated exposure	Category 1
(Nervous system, Respiratory organ)	

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ENVIRONMENTAL HAZARDS

Aquatic toxicity (chronic)	Category 4

Hazard is not stated, the classification can not be classified or exempt.

GHS LABEL ELEMENTS INCLUDING PRECAUTIONARY STATEMENTS

SYMBOL

SIGNAL WORD : DANGER

HAZARD STATEMENT

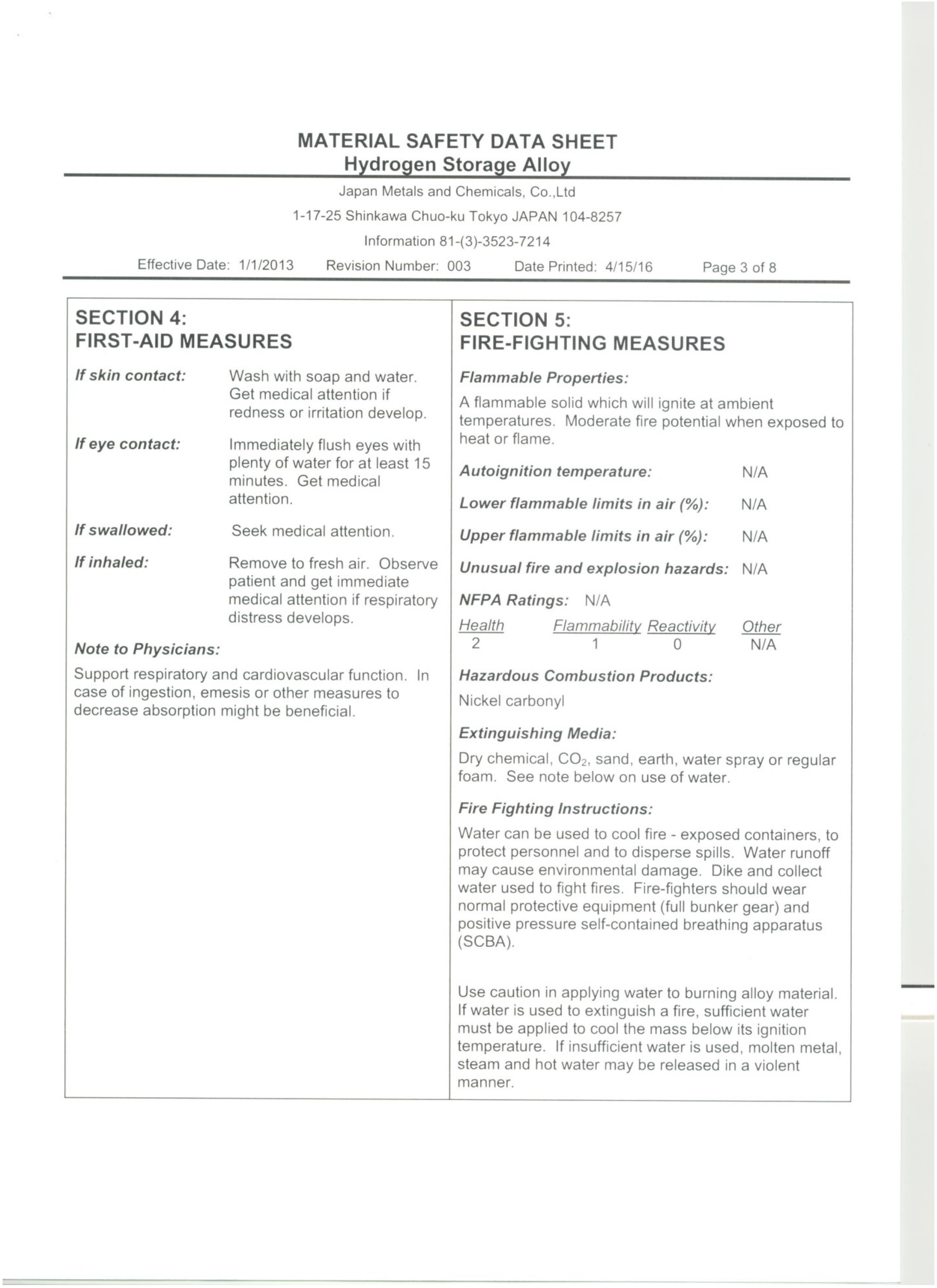
- Causes mild skin irritation.
- Causes eye irritation.
- May causes allergy or asthma symptoms or breathing difficulties if inhaled.
- May cause allergic skin reaction.
- Suspected of carcinogenic potential.
- May damage fertility or the unborn child
- Causes damage to Respiratory, nervous system, kidneys, liver, heart.
- May cause respiratory irritation
- Causes damage to nervous system, respiratory disease thorough prolonged or repeated exposure.
- Toxic to aquatic life with long lasting effects

SECTION 3: COMPOSITION/INFORMATION ON INGREDIENTS

Chemical Name: N/A ***CAS #:*** N/A ***%Wt.:*** N/A

Ingredient	*CAS#*	*OSHA PEL TWA*	*OSHA PEL STEL*	*ACGIH TLV TWA*	*ACGIH TLV STEL*	*%Wt*
Nickel	7440-02-0	1 mg/m^3	-	1 mg/m^3	-	45-68
Aluminum	7429-90-5	15	-	10	-	0.5-7
Lanthanum	7439-91-0	N/A	-	N/A	-	0-41
Cerium	7440-45-1	N/A	-	N/A	-	0-5
Praseodymium	—	N/A	-	N/A	-	0-5
Neodymium	7440-00-8	N/A	-	N/A	-	0-5
Samarium	7440-19-9	N/A	-	N/A	-	0-5

N/A: not applicable (C): ceiling limit TWA: 8-hour time weighted average
mg/m^3: milligrams per cubic meter of air
Note: Some State OSHA programs may enforce limits different from Federal OSHA.



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Information 81-(3)-3523-7214

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SECTION 4:
FIRST-AID MEASURES

If skin contact:	Wash with soap and water. Get medical attention if redness or irritation develop.
If eye contact:	Immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention.
If swallowed:	Seek medical attention.
If inhaled:	Remove to fresh air. Observe patient and get immediate medical attention if respiratory distress develops.

Note to Physicians:

Support respiratory and cardiovascular function. In case of ingestion, emesis or other measures to decrease absorption might be beneficial.

SECTION 5:
FIRE-FIGHTING MEASURES

Flammable Properties:

A flammable solid which will ignite at ambient temperatures. Moderate fire potential when exposed to heat or flame.

Autoignition temperature:	N/A
Lower flammable limits in air (%):	N/A
Upper flammable limits in air (%):	N/A
Unusual fire and explosion hazards:	N/A

NFPA Ratings: N/A

Health	*Flammability*	*Reactivity*	*Other*
2	1	0	N/A

Hazardous Combustion Products:

Nickel carbonyl

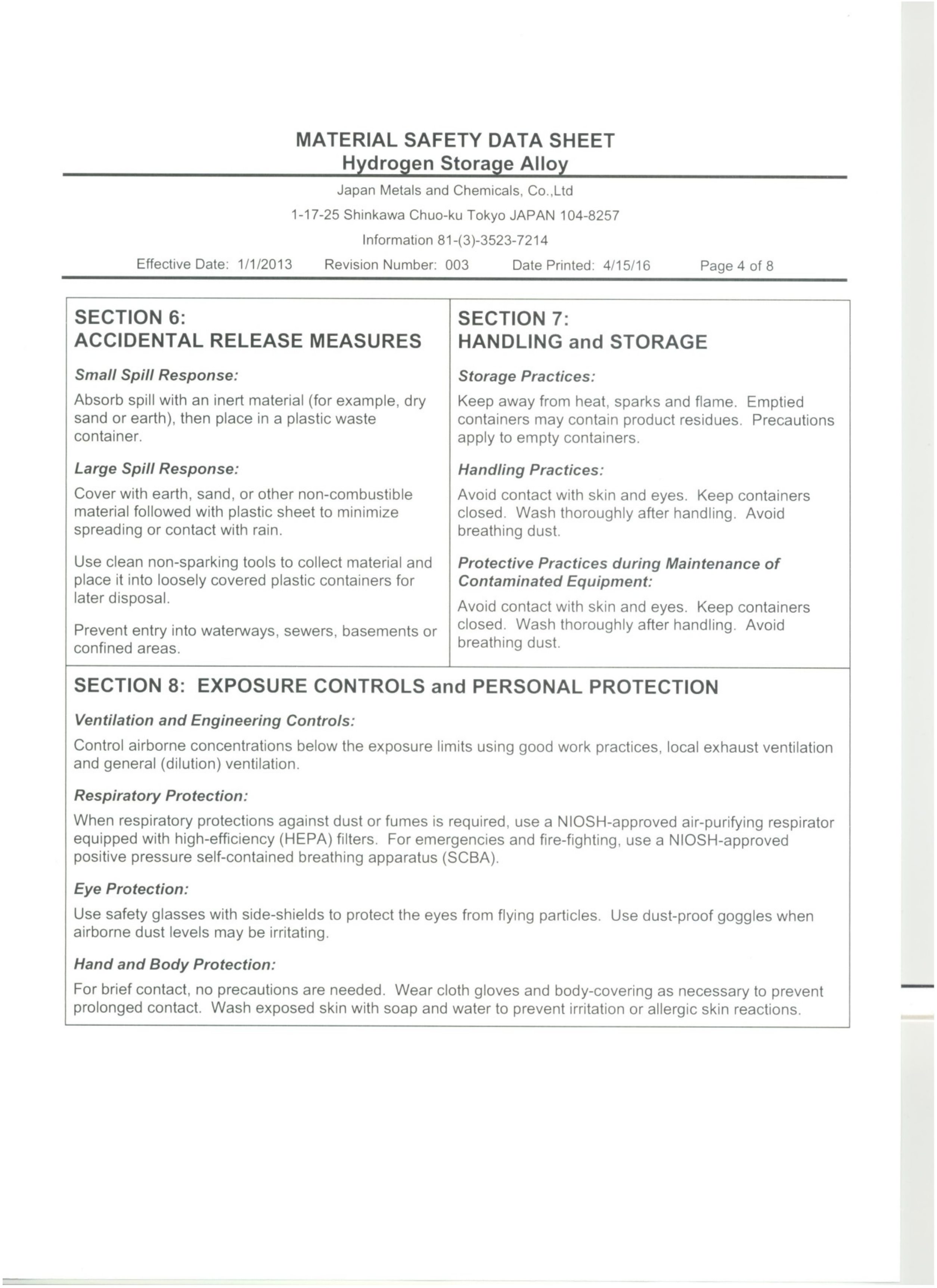
Extinguishing Media:

Dry chemical, CO_2, sand, earth, water spray or regular foam. See note below on use of water.

Fire Fighting Instructions:

Water can be used to cool fire - exposed containers, to protect personnel and to disperse spills. Water runoff may cause environmental damage. Dike and collect water used to fight fires. Fire-fighters should wear normal protective equipment (full bunker gear) and positive pressure self-contained breathing apparatus (SCBA).

Use caution in applying water to burning alloy material. If water is used to extinguish a fire, sufficient water must be applied to cool the mass below its ignition temperature. If insufficient water is used, molten metal, steam and hot water may be released in a violent manner.



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SECTION 6:
ACCIDENTAL RELEASE MEASURES

Small Spill Response:

Absorb spill with an inert material (for example, dry sand or earth), then place in a plastic waste container.

Large Spill Response:

Cover with earth, sand, or other non-combustible material followed with plastic sheet to minimize spreading or contact with rain.

Use clean non-sparking tools to collect material and place it into loosely covered plastic containers for later disposal.

Prevent entry into waterways, sewers, basements or confined areas.

SECTION 7:
HANDLING and STORAGE

Storage Practices:

Keep away from heat, sparks and flame. Emptied containers may contain product residues. Precautions apply to empty containers.

Handling Practices:

Avoid contact with skin and eyes. Keep containers closed. Wash thoroughly after handling. Avoid breathing dust.

Protective Practices during Maintenance of Contaminated Equipment:

Avoid contact with skin and eyes. Keep containers closed. Wash thoroughly after handling. Avoid breathing dust.

SECTION 8: EXPOSURE CONTROLS and PERSONAL PROTECTION

Ventilation and Engineering Controls:

Control airborne concentrations below the exposure limits using good work practices, local exhaust ventilation and general (dilution) ventilation.

Respiratory Protection:

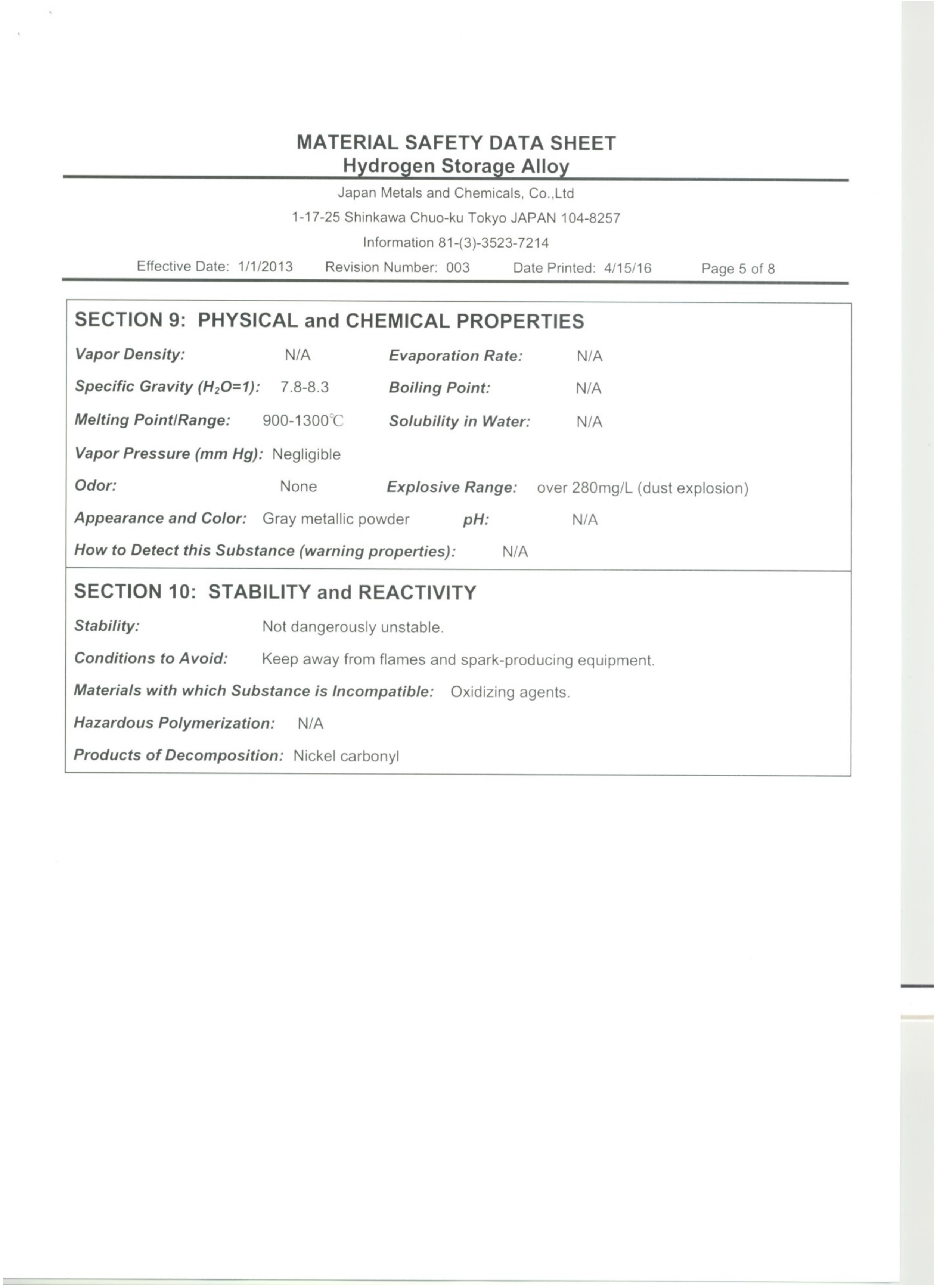
When respiratory protections against dust or fumes is required, use a NIOSH-approved air-purifying respirator equipped with high-efficiency (HEPA) filters. For emergencies and fire-fighting, use a NIOSH-approved positive pressure self-contained breathing apparatus (SCBA).

Eye Protection:

Use safety glasses with side-shields to protect the eyes from flying particles. Use dust-proof goggles when airborne dust levels may be irritating.

Hand and Body Protection:

For brief contact, no precautions are needed. Wear cloth gloves and body-covering as necessary to prevent prolonged contact. Wash exposed skin with soap and water to prevent irritation or allergic skin reactions.



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Information 81-(3)-3523-7214

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SECTION 9: PHYSICAL and CHEMICAL PROPERTIES

Vapor Density: N/A ***Evaporation Rate:*** N/A

Specific Gravity (H_2O=1): 7.8-8.3 ***Boiling Point:*** N/A

Melting Point/Range: 900-1300℃ ***Solubility in Water:*** N/A

Vapor Pressure (mm Hg): Negligible

Odor: None ***Explosive Range:*** over 280mg/L (dust explosion)

Appearance and Color: Gray metallic powder ***pH:*** N/A

How to Detect this Substance (warning properties): N/A

SECTION 10: STABILITY and REACTIVITY

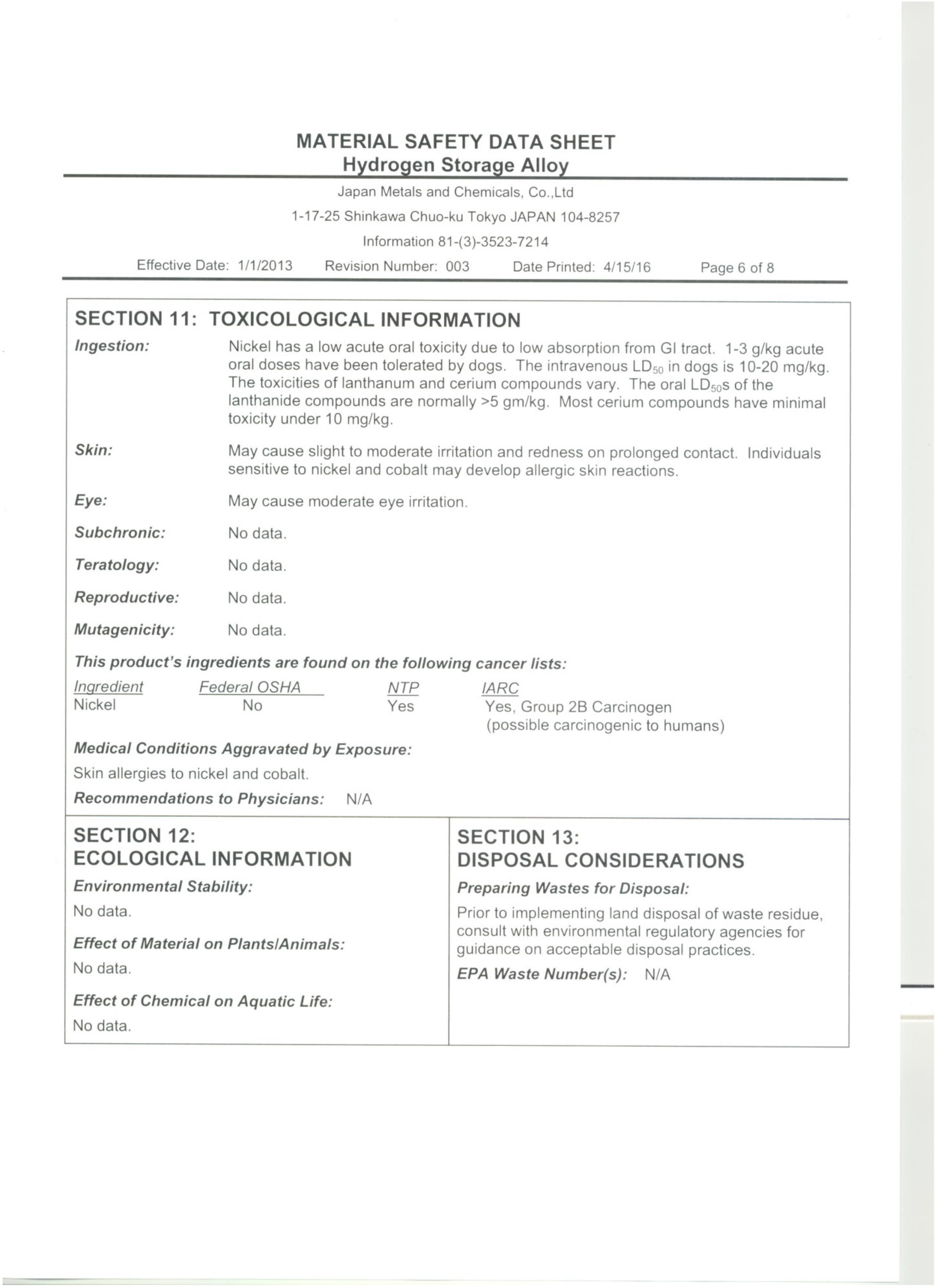
Stability: Not dangerously unstable.

Conditions to Avoid: Keep away from flames and spark-producing equipment.

Materials with which Substance is Incompatible: Oxidizing agents.

Hazardous Polymerization: N/A

Products of Decomposition: Nickel carbonyl



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Information 81-(3)-3523-7214

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SECTION 11: TOXICOLOGICAL INFORMATION

Ingestion: Nickel has a low acute oral toxicity due to low absorption from GI tract. 1-3 g/kg acute oral doses have been tolerated by dogs. The intravenous LD_{50} in dogs is 10-20 mg/kg. The toxicities of lanthanum and cerium compounds vary. The oral LD_{50}s of the lanthanide compounds are normally >5 gm/kg. Most cerium compounds have minimal toxicity under 10 mg/kg.

Skin: May cause slight to moderate irritation and redness on prolonged contact. Individuals sensitive to nickel and cobalt may develop allergic skin reactions.

Eye: May cause moderate eye irritation.

Subchronic: No data.

Teratology: No data.

Reproductive: No data.

Mutagenicity: No data.

This product's ingredients are found on the following cancer lists:

Ingredient	*Federal OSHA*	*NTP*	*IARC*
Nickel	No	Yes	Yes, Group 2B Carcinogen (possible carcinogenic to humans)

Medical Conditions Aggravated by Exposure:

Skin allergies to nickel and cobalt.

Recommendations to Physicians: N/A

SECTION 12: ECOLOGICAL INFORMATION

Environmental Stability:

No data.

Effect of Material on Plants/Animals:

No data.

Effect of Chemical on Aquatic Life:

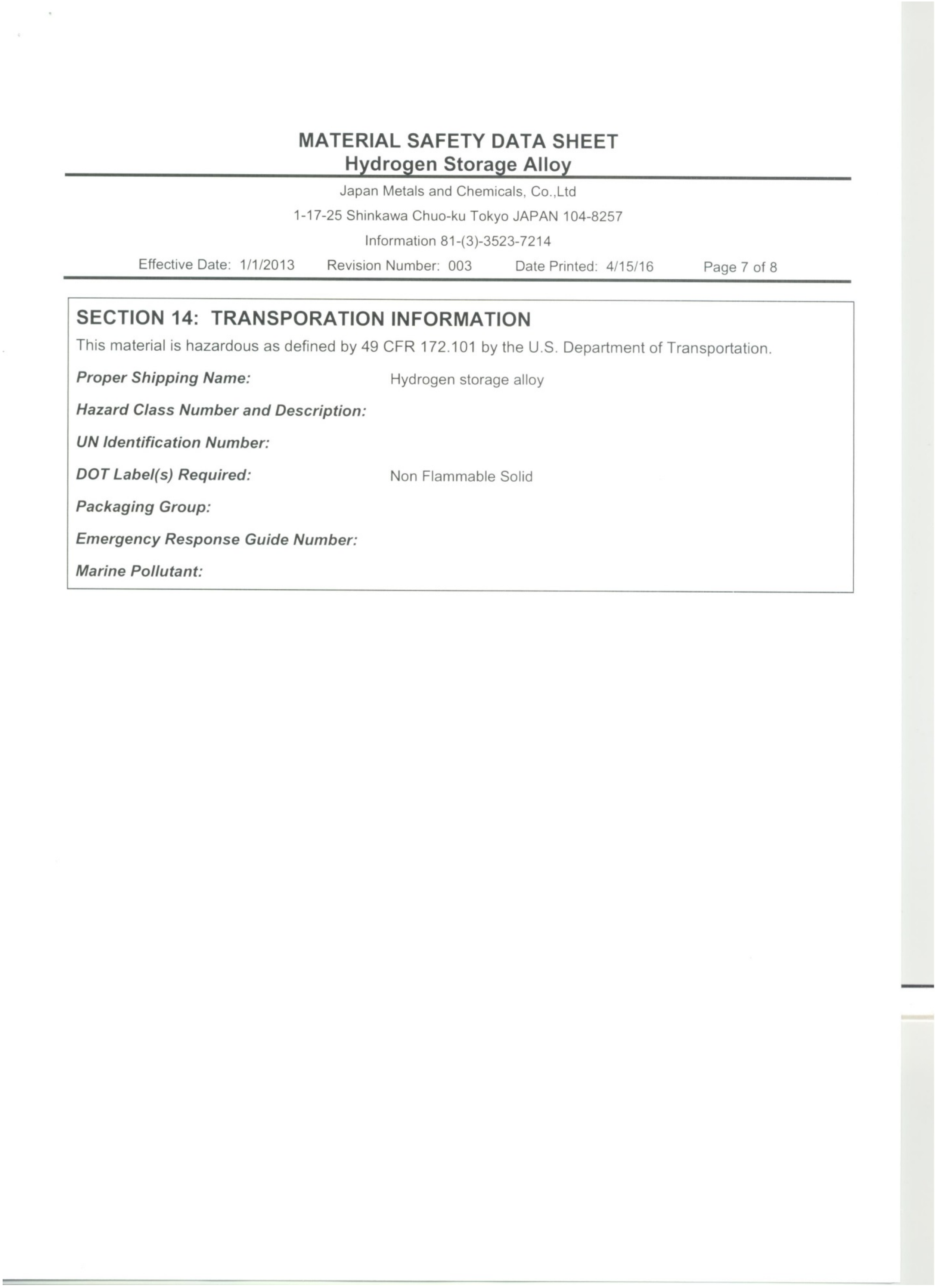
No data.

SECTION 13: DISPOSAL CONSIDERATIONS

Preparing Wastes for Disposal:

Prior to implementing land disposal of waste residue, consult with environmental regulatory agencies for guidance on acceptable disposal practices.

EPA Waste Number(s): N/A



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SECTION 14: TRANSPORATION INFORMATION

This material is hazardous as defined by 49 CFR 172.101 by the U.S. Department of Transportation.

Proper Shipping Name: Hydrogen storage alloy

Hazard Class Number and Description:

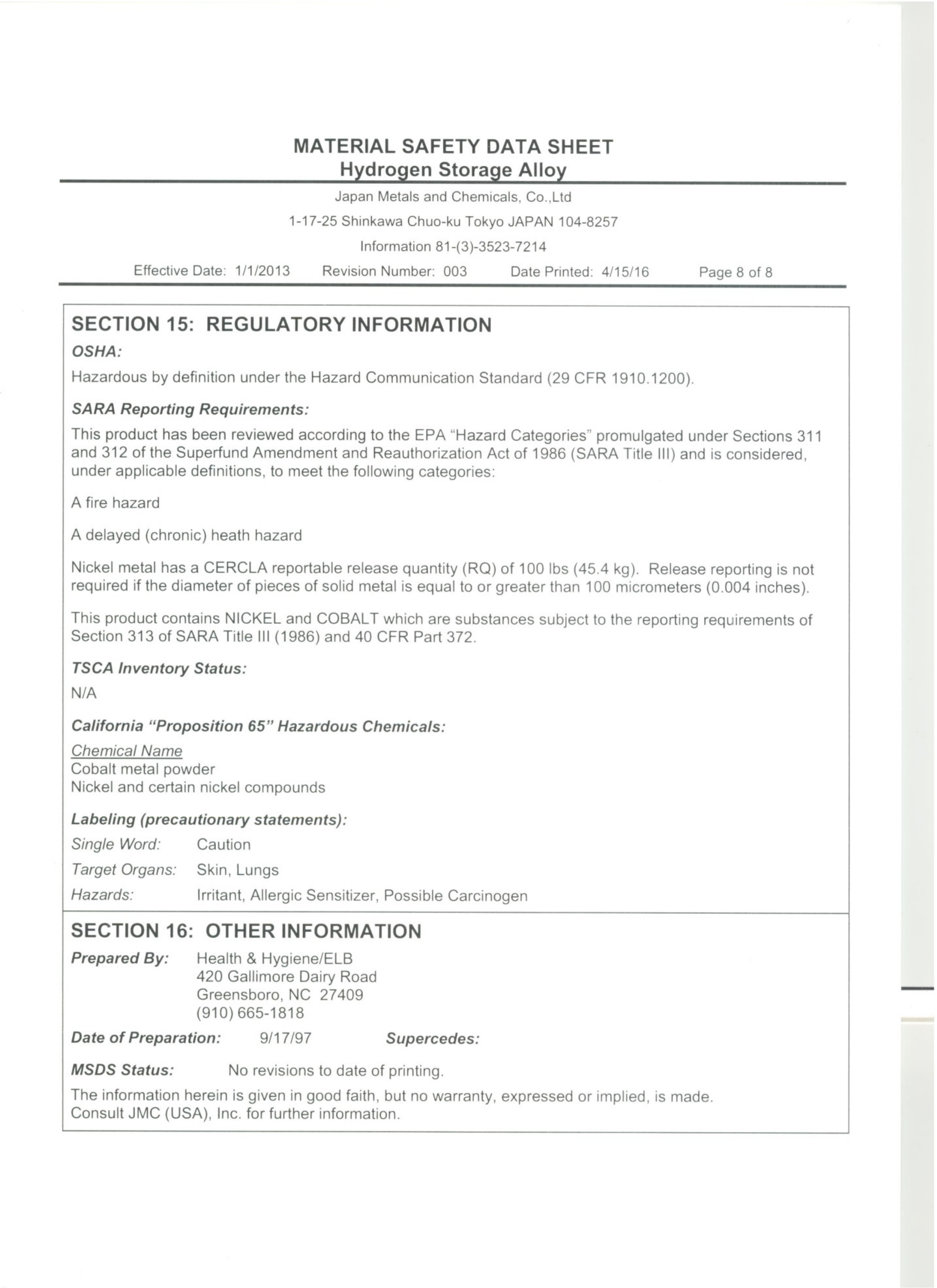
UN Identification Number:

DOT Label(s) Required: Non Flammable Solid

Packaging Group:

Emergency Response Guide Number:

Marine Pollutant:



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SECTION 15: REGULATORY INFORMATION

OSHA:

Hazardous by definition under the Hazard Communication Standard (29 CFR 1910.1200).

SARA Reporting Requirements:

This product has been reviewed according to the EPA "Hazard Categories" promulgated under Sections 311 and 312 of the Superfund Amendment and Reauthorization Act of 1986 (SARA Title III) and is considered, under applicable definitions, to meet the following categories:

A fire hazard

A delayed (chronic) heath hazard

Nickel metal has a CERCLA reportable release quantity (RQ) of 100 lbs (45.4 kg). Release reporting is not required if the diameter of pieces of solid metal is equal to or greater than 100 micrometers (0.004 inches).

This product contains NICKEL and COBALT which are substances subject to the reporting requirements of Section 313 of SARA Title III (1986) and 40 CFR Part 372.

TSCA Inventory Status:

N/A

California "Proposition 65" Hazardous Chemicals:

Chemical Name
Cobalt metal powder
Nickel and certain nickel compounds

Labeling (precautionary statements):

Single Word:	Caution
Target Organs:	Skin, Lungs
Hazards:	Irritant, Allergic Sensitizer, Possible Carcinogen

SECTION 16: OTHER INFORMATION

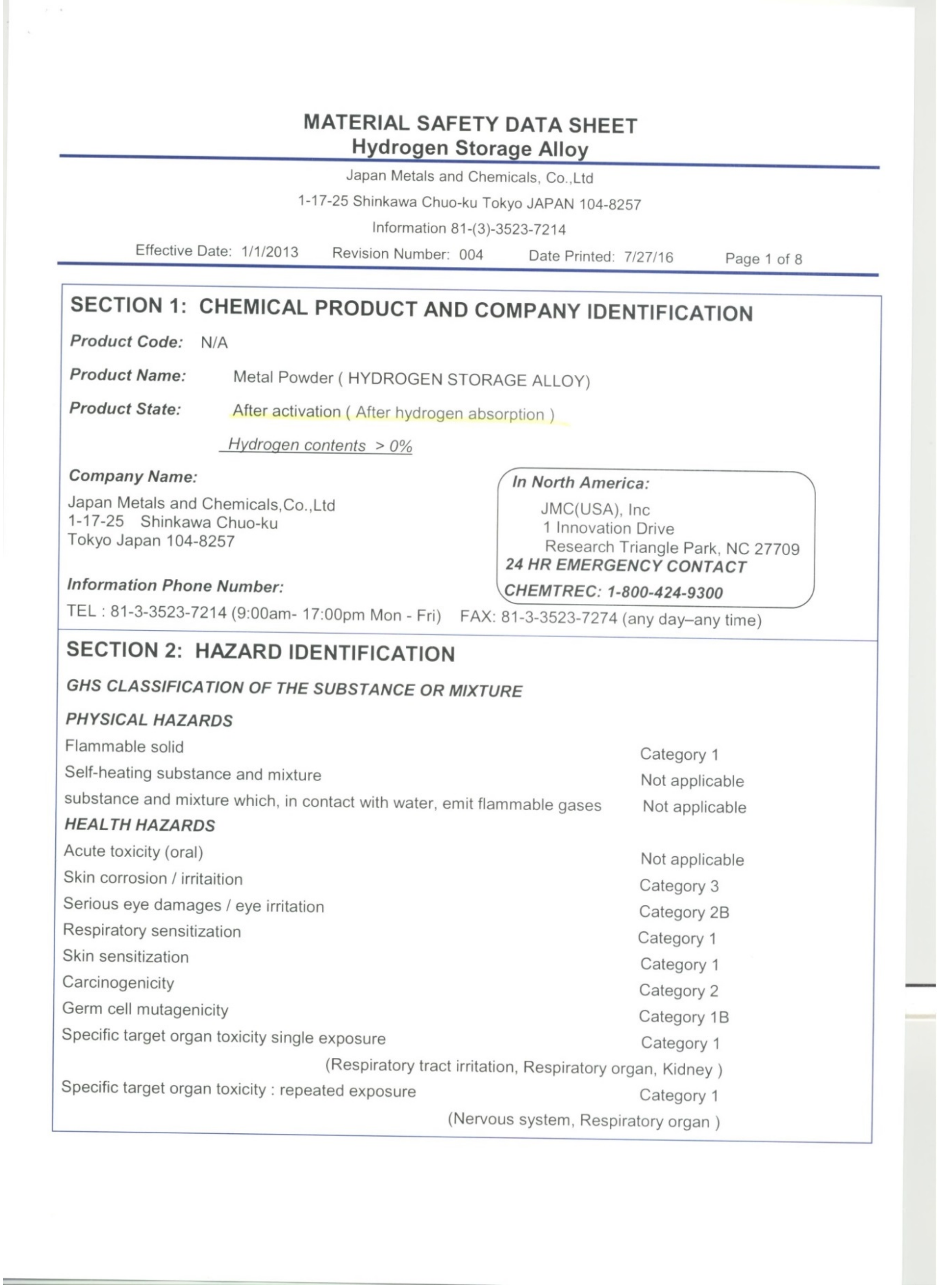
Prepared By:
Health & Hygiene/ELB
420 Gallimore Dairy Road
Greensboro, NC 27409
(910) 665-1818

Date of Preparation: 9/17/97 ***Supercedes:***

MSDS Status: No revisions to date of printing.

The information herein is given in good faith, but no warranty, expressed or implied, is made. Consult JMC (USA), Inc. for further information.

Part 3. SDS – activated material (either LANA75 or LANA85)



MATERIAL SAFETY DATA SHEET
Hydrogen Storage Alloy

Japan Metals and Chemicals, Co.,Ltd
1-17-25 Shinkawa Chuo-ku Tokyo JAPAN 104-8257
Information 81-(3)-3523-7214

Effective Date: 1/1/2013 Revision Number: 004 Date Printed: 7/27/16 Page 1 of 8

SECTION 1: CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Code: N/A

Product Name: Metal Powder (HYDROGEN STORAGE ALLOY)

Product State: After activation (After hydrogen absorption)

Hydrogen contents > 0%

Company Name:

Japan Metals and Chemicals,Co.,Ltd
1-17-25 Shinkawa Chuo-ku
Tokyo Japan 104-8257

In North America:

JMC(USA), Inc
1 Innovation Drive
Research Triangle Park, NC 27709

24 HR EMERGENCY CONTACT

CHEMTREC: 1-800-424-9300

Information Phone Number:

TEL : 81-3-3523-7214 (9:00am- 17:00pm Mon - Fri) FAX: 81-3-3523-7274 (any day–any time)

SECTION 2: HAZARD IDENTIFICATION

GHS CLASSIFICATION OF THE SUBSTANCE OR MIXTURE

PHYSICAL HAZARDS

Flammable solid	Category 1
Self-heating substance and mixture	Not applicable
substance and mixture which, in contact with water, emit flammable gases	Not applicable

HEALTH HAZARDS

Acute toxicity (oral)	Not applicable
Skin corrosion / irritaition	Category 3
Serious eye damages / eye irritation	Category 2B
Respiratory sensitization	Category 1
Skin sensitization	Category 1
Carcinogenicity	Category 2
Germ cell mutagenicity	Category 1B
Specific target organ toxicity single exposure	Category 1 (Respiratory tract irritation, Respiratory organ, Kidney)
Specific target organ toxicity : repeated exposure	Category 1 (Nervous system, Respiratory organ)

ENVIRONMENTAL HAZARDS

Aquatic toxicity (chronic)	Category 4

Hazard is not stated, the classification can not be classified or exempt.

GHS LABEL ELEMENTS INCLUDING PRECAUTIONARY STATEMENTS

SYMBOL

SIGNAL WORD : DANGER

HAZARD STATEMENT

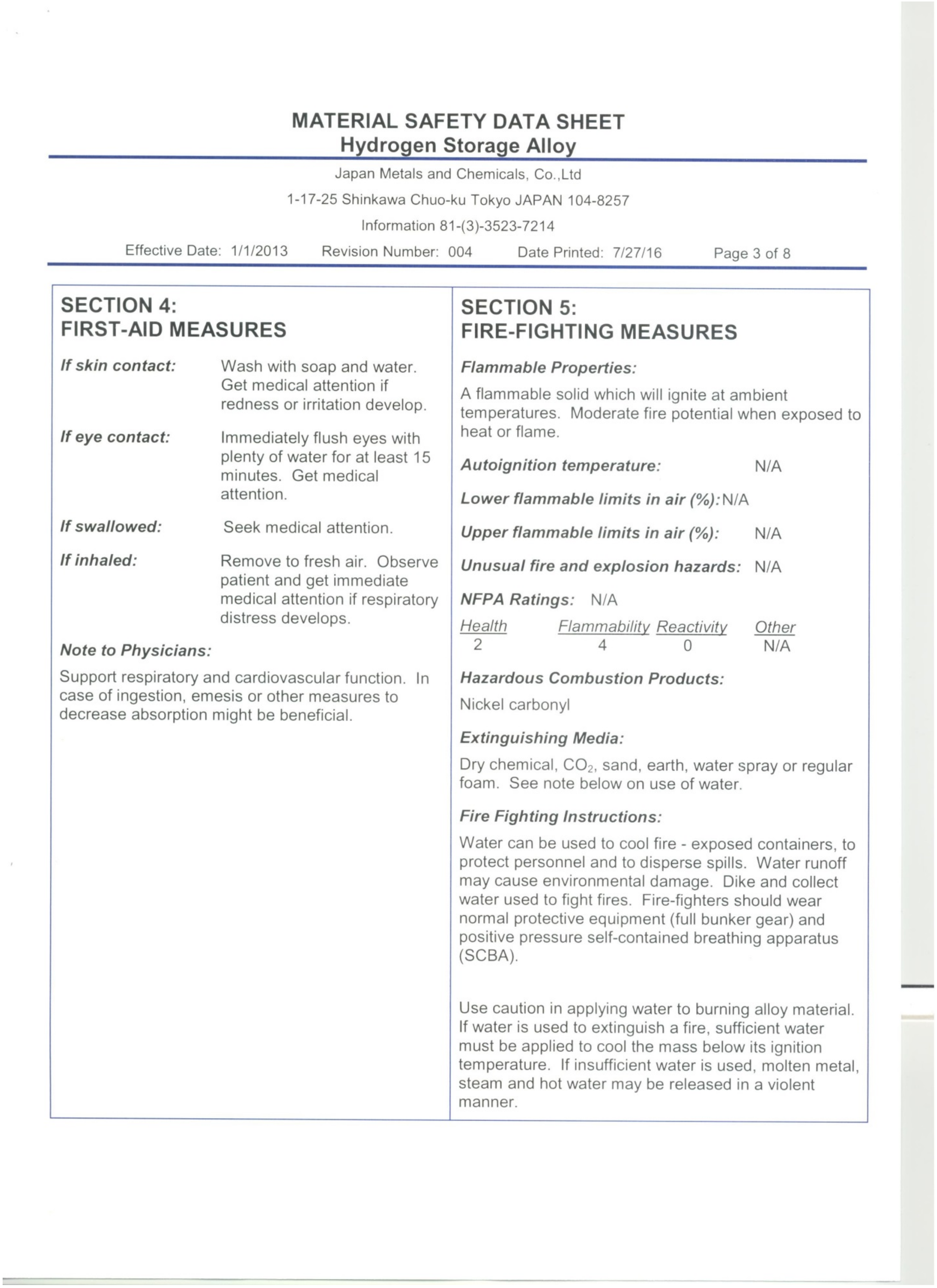
- Flammable solids, highly flammable dry powder
- Causes mild skin irritation.
- Causes eye irritation.
- May causes allergy or asthma symptoms or breathing difficulties if inhaled.
- May cause allergic skin reaction.
- Suspected of carcinogenic potential.
- May damage fertility or the unborn child
- Causes damage to Respiratory, nervous system, kidneys, liver, heart.
- May cause respiratory irritation
- Causes damage to nervous system, respiratory disease thorough prolonged or repeated exposure.
- Toxic to aquatic life with long lasting effects

SECTION 3: COMPOSITION/INFORMATION ON INGREDIENTS

Chemical Name: N/A ***CAS #:*** N/A ***%Wt.:*** N/A

Ingredient	*CAS#*	*OSHA PEL TWA*	*OSHA PEL STEL*	*ACGIH TLV TWA*	*ACGIH TLV STEL*	*%Wt*
Nickel	7440-02-0	1 mg/m^3	-	1 mg/m^3	-	45-68
Aluminum	7429-90-5	15	-	10	-	0.5-7
Lanthanum	7439-91-0	N/A	-	N/A	-	0-41
Cerium	7440-45-1	N/A	-	N/A	-	0-5
Praseodymium	—	N/A	-	N/A	-	0-5
Neodymium	7440-00-8	N/A	-	N/A	-	0-5
Samarium	7440-19-9	N/A	-	N/A	-	0-5

N/A: not applicable (C): ceiling limit TWA: 8-hour time weighted average
mg/m^3: milligrams per cubic meter of air
Note: Some State OSHA programs may enforce limits different from Federal OSHA.



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Effective Date: 1/1/2013 Revision Number: 004 Date Printed: 7/27/16 Page 3 of 8

SECTION 4:
FIRST-AID MEASURES

If skin contact:	Wash with soap and water. Get medical attention if redness or irritation develop.
If eye contact:	Immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention.
If swallowed:	Seek medical attention.
If inhaled:	Remove to fresh air. Observe patient and get immediate medical attention if respiratory distress develops.

Note to Physicians:

Support respiratory and cardiovascular function. In case of ingestion, emesis or other measures to decrease absorption might be beneficial.

SECTION 5:
FIRE-FIGHTING MEASURES

Flammable Properties:

A flammable solid which will ignite at ambient temperatures. Moderate fire potential when exposed to heat or flame.

Autoignition temperature: N/A

Lower flammable limits in air (%): N/A

Upper flammable limits in air (%): N/A

Unusual fire and explosion hazards: N/A

NFPA Ratings: N/A

Health	*Flammability*	*Reactivity*	*Other*
2	4	0	N/A

Hazardous Combustion Products:

Nickel carbonyl

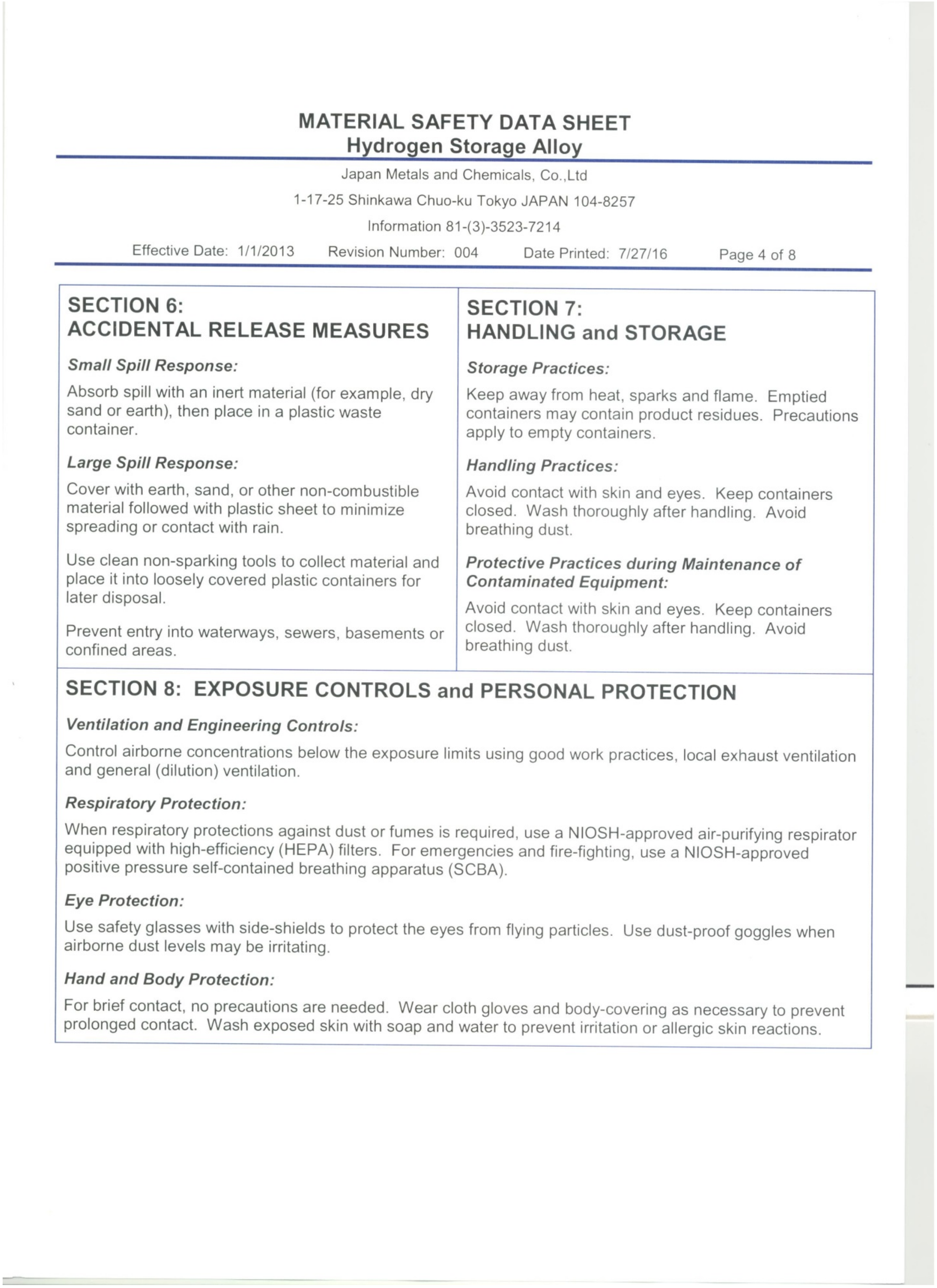
Extinguishing Media:

Dry chemical, CO_2, sand, earth, water spray or regular foam. See note below on use of water.

Fire Fighting Instructions:

Water can be used to cool fire - exposed containers, to protect personnel and to disperse spills. Water runoff may cause environmental damage. Dike and collect water used to fight fires. Fire-fighters should wear normal protective equipment (full bunker gear) and positive pressure self-contained breathing apparatus (SCBA).

Use caution in applying water to burning alloy material. If water is used to extinguish a fire, sufficient water must be applied to cool the mass below its ignition temperature. If insufficient water is used, molten metal, steam and hot water may be released in a violent manner.



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SECTION 6:
ACCIDENTAL RELEASE MEASURES

Small Spill Response:

Absorb spill with an inert material (for example, dry sand or earth), then place in a plastic waste container.

Large Spill Response:

Cover with earth, sand, or other non-combustible material followed with plastic sheet to minimize spreading or contact with rain.

Use clean non-sparking tools to collect material and place it into loosely covered plastic containers for later disposal.

Prevent entry into waterways, sewers, basements or confined areas.

SECTION 7:
HANDLING and STORAGE

Storage Practices:

Keep away from heat, sparks and flame. Emptied containers may contain product residues. Precautions apply to empty containers.

Handling Practices:

Avoid contact with skin and eyes. Keep containers closed. Wash thoroughly after handling. Avoid breathing dust.

Protective Practices during Maintenance of Contaminated Equipment:

Avoid contact with skin and eyes. Keep containers closed. Wash thoroughly after handling. Avoid breathing dust.

SECTION 8: EXPOSURE CONTROLS and PERSONAL PROTECTION

Ventilation and Engineering Controls:

Control airborne concentrations below the exposure limits using good work practices, local exhaust ventilation and general (dilution) ventilation.

Respiratory Protection:

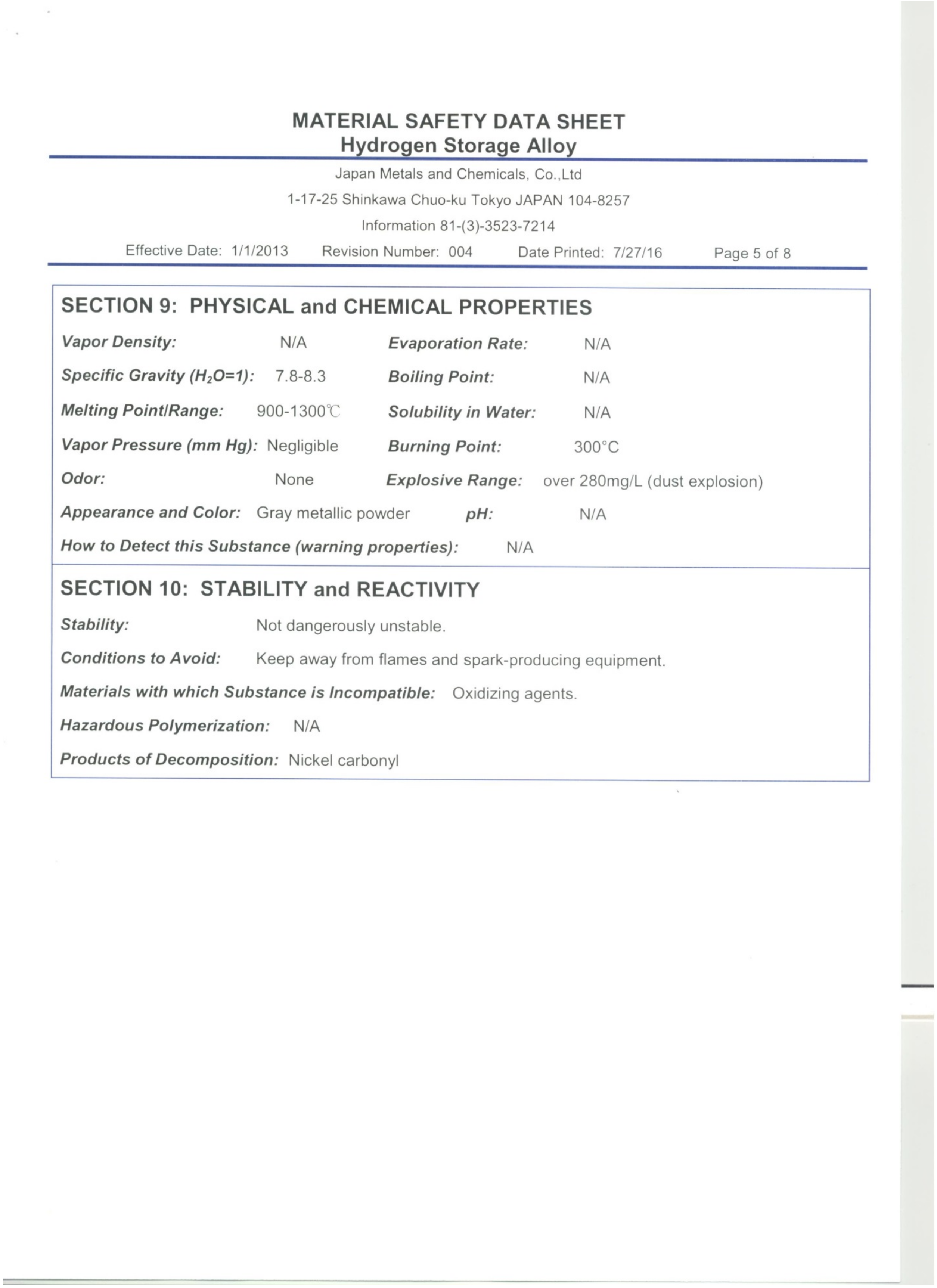
When respiratory protections against dust or fumes is required, use a NIOSH-approved air-purifying respirator equipped with high-efficiency (HEPA) filters. For emergencies and fire-fighting, use a NIOSH-approved positive pressure self-contained breathing apparatus (SCBA).

Eye Protection:

Use safety glasses with side-shields to protect the eyes from flying particles. Use dust-proof goggles when airborne dust levels may be irritating.

Hand and Body Protection:

For brief contact, no precautions are needed. Wear cloth gloves and body-covering as necessary to prevent prolonged contact. Wash exposed skin with soap and water to prevent irritation or allergic skin reactions.



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SECTION 9: PHYSICAL and CHEMICAL PROPERTIES

Vapor Density:	N/A	***Evaporation Rate:***	N/A
Specific Gravity (H_2O=1):	7.8-8.3	***Boiling Point:***	N/A
Melting Point/Range:	900-1300℃	***Solubility in Water:***	N/A
Vapor Pressure (mm Hg):	Negligible	***Burning Point:***	300°C
Odor:	None	***Explosive Range:***	over 280mg/L (dust explosion)
Appearance and Color:	Gray metallic powder	***pH:***	N/A

How to Detect this Substance (warning properties): N/A

SECTION 10: STABILITY and REACTIVITY

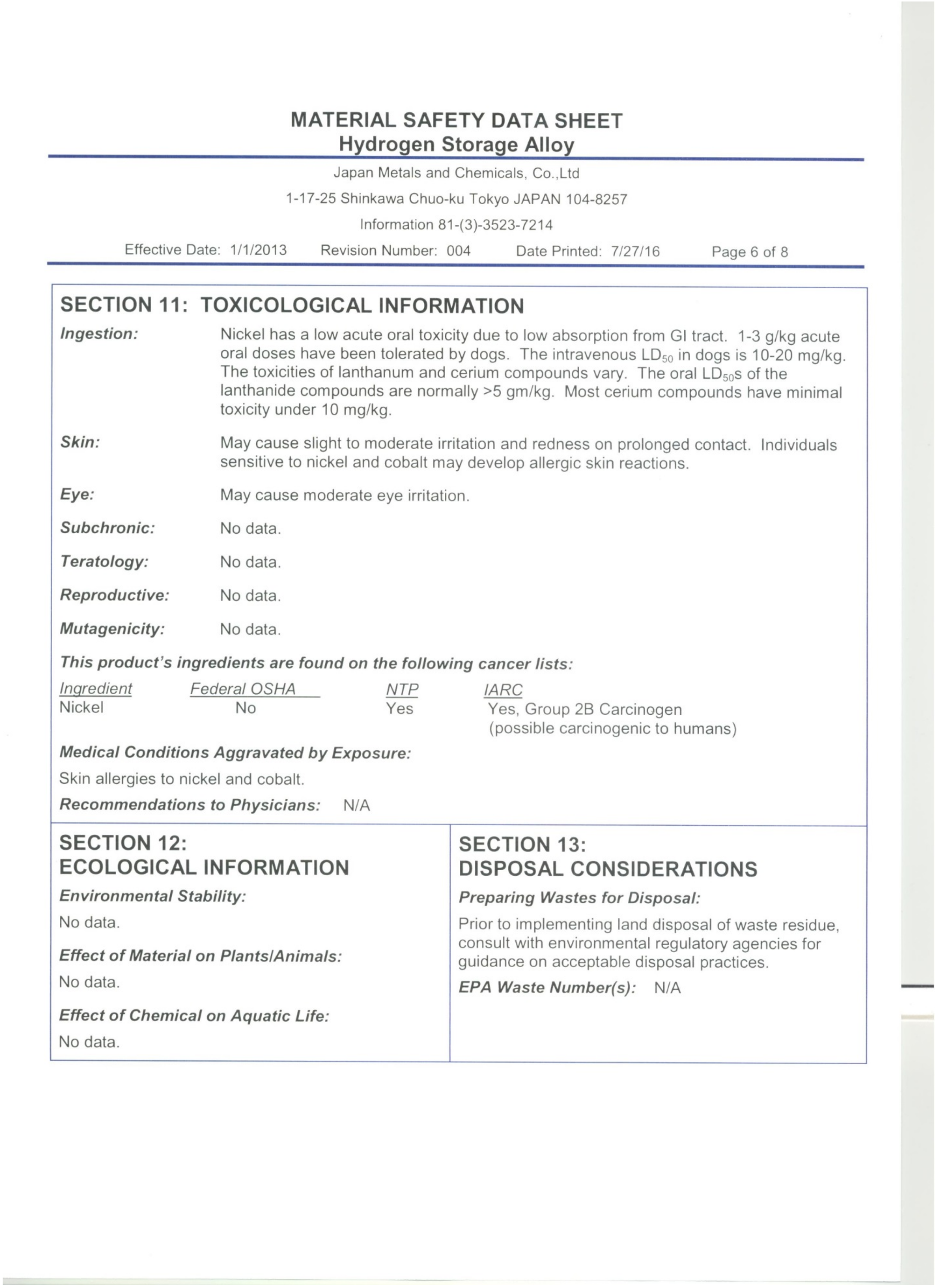
Stability: Not dangerously unstable.

Conditions to Avoid: Keep away from flames and spark-producing equipment.

Materials with which Substance is Incompatible: Oxidizing agents.

Hazardous Polymerization: N/A

Products of Decomposition: Nickel carbonyl



MATERIAL SAFETY DATA SHEET
Hydrogen Storage Alloy

Japan Metals and Chemicals, Co.,Ltd
1-17-25 Shinkawa Chuo-ku Tokyo JAPAN 104-8257
Information 81-(3)-3523-7214

Effective Date: 1/1/2013 Revision Number: 004 Date Printed: 7/27/16 Page 6 of 8

SECTION 11: TOXICOLOGICAL INFORMATION

Ingestion: Nickel has a low acute oral toxicity due to low absorption from GI tract. 1-3 g/kg acute oral doses have been tolerated by dogs. The intravenous LD_{50} in dogs is 10-20 mg/kg. The toxicities of lanthanum and cerium compounds vary. The oral LD_{50}s of the lanthanide compounds are normally >5 gm/kg. Most cerium compounds have minimal toxicity under 10 mg/kg.

Skin: May cause slight to moderate irritation and redness on prolonged contact. Individuals sensitive to nickel and cobalt may develop allergic skin reactions.

Eye: May cause moderate eye irritation.

Subchronic: No data.

Teratology: No data.

Reproductive: No data.

Mutagenicity: No data.

This product's ingredients are found on the following cancer lists:

Ingredient	*Federal OSHA*	*NTP*	*IARC*
Nickel	No	Yes	Yes, Group 2B Carcinogen (possible carcinogenic to humans)

Medical Conditions Aggravated by Exposure:

Skin allergies to nickel and cobalt.

Recommendations to Physicians: N/A

SECTION 12: ECOLOGICAL INFORMATION

Environmental Stability:

No data.

Effect of Material on Plants/Animals:

No data.

Effect of Chemical on Aquatic Life:

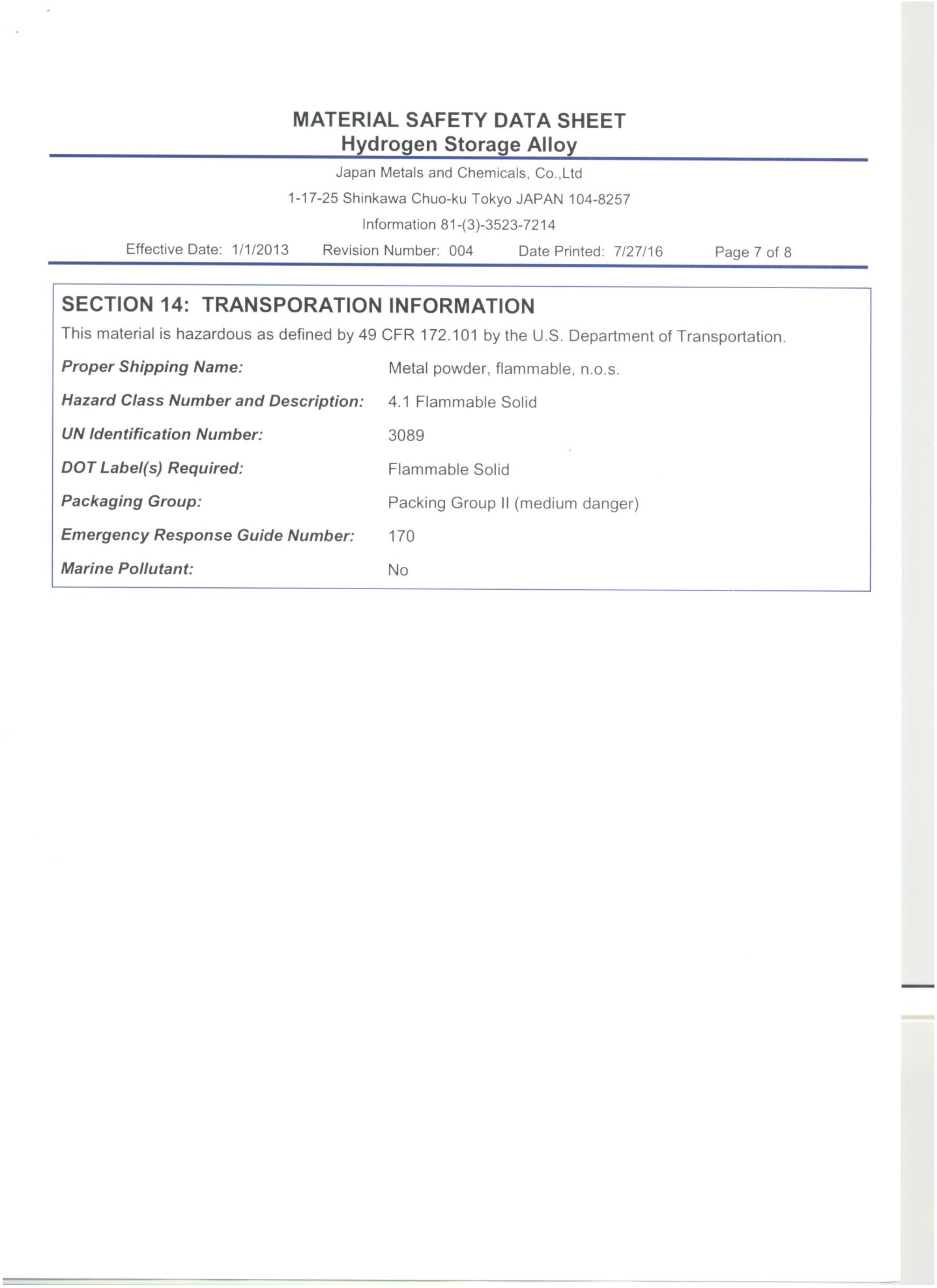
No data.

SECTION 13: DISPOSAL CONSIDERATIONS

Preparing Wastes for Disposal:

Prior to implementing land disposal of waste residue, consult with environmental regulatory agencies for guidance on acceptable disposal practices.

EPA Waste Number(s): N/A



MATERIAL SAFETY DATA SHEET
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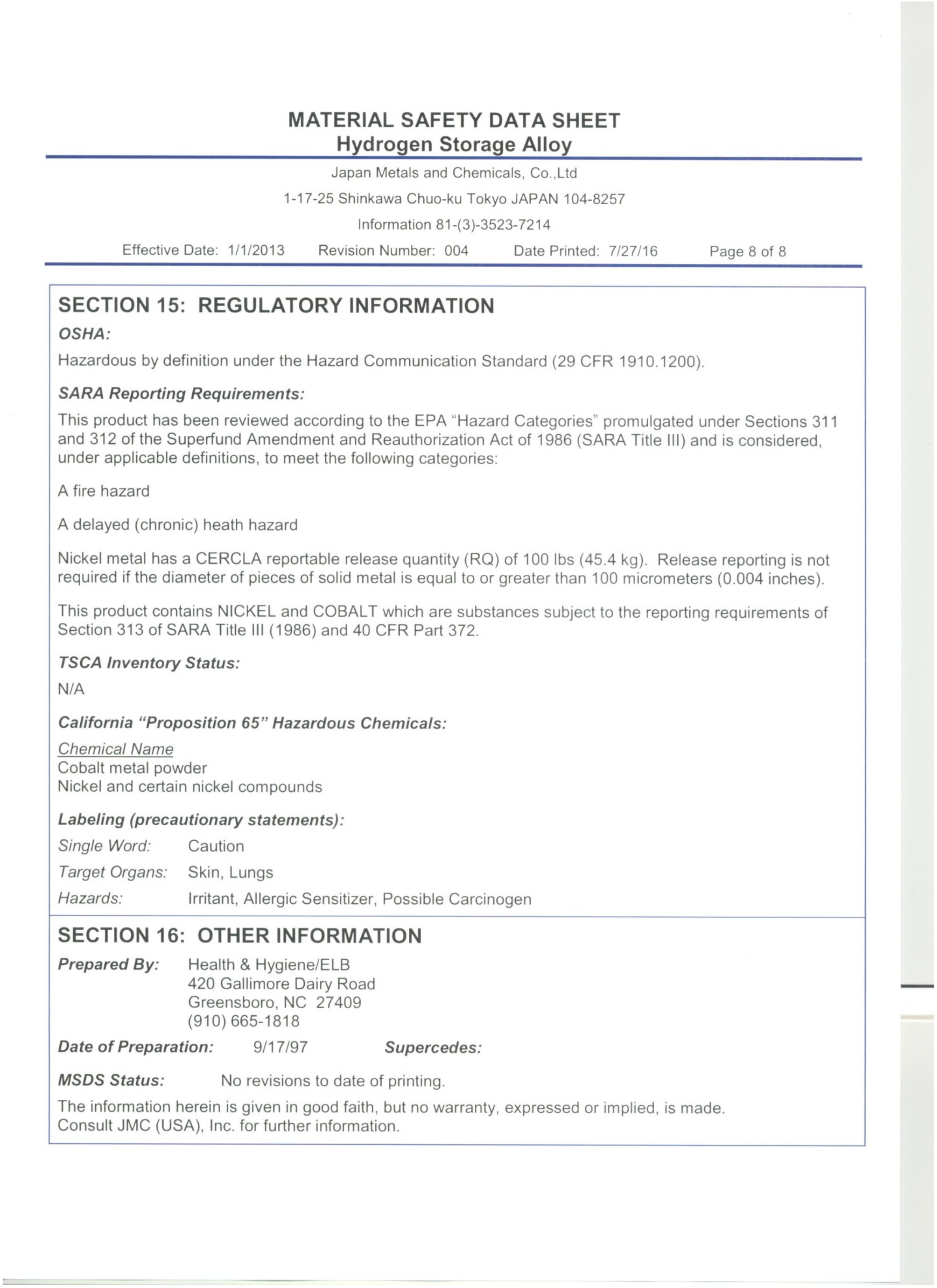
Effective Date: 1/1/2013 Revision Number: 004 Date Printed: 7/27/16 Page 7 of 8

SECTION 14: TRANSPORATION INFORMATION

This material is hazardous as defined by 49 CFR 172.101 by the U.S. Department of Transportation.

Proper Shipping Name:	Metal powder, flammable, n.o.s.
Hazard Class Number and Description:	4.1 Flammable Solid
UN Identification Number:	3089
DOT Label(s) Required:	Flammable Solid
Packaging Group:	Packing Group II (medium danger)
Emergency Response Guide Number:	170
Marine Pollutant:	No



MATERIAL SAFETY DATA SHEET
Hydrogen Storage Alloy

Japan Metals and Chemicals, Co.,Ltd
1-17-25 Shinkawa Chuo-ku Tokyo JAPAN 104-8257
Information 81-(3)-3523-7214

Effective Date: 1/1/2013 Revision Number: 004 Date Printed: 7/27/16 Page 8 of 8

SECTION 15: REGULATORY INFORMATION

OSHA:

Hazardous by definition under the Hazard Communication Standard (29 CFR 1910.1200).

SARA Reporting Requirements:

This product has been reviewed according to the EPA "Hazard Categories" promulgated under Sections 311 and 312 of the Superfund Amendment and Reauthorization Act of 1986 (SARA Title III) and is considered, under applicable definitions, to meet the following categories:

A fire hazard

A delayed (chronic) heath hazard

Nickel metal has a CERCLA reportable release quantity (RQ) of 100 lbs (45.4 kg). Release reporting is not required if the diameter of pieces of solid metal is equal to or greater than 100 micrometers (0.004 inches).

This product contains NICKEL and COBALT which are substances subject to the reporting requirements of Section 313 of SARA Title III (1986) and 40 CFR Part 372.

TSCA Inventory Status:

N/A

California "Proposition 65" Hazardous Chemicals:

Chemical Name
Cobalt metal powder
Nickel and certain nickel compounds

Labeling (precautionary statements):

Single Word: Caution

Target Organs: Skin, Lungs

Hazards: Irritant, Allergic Sensitizer, Possible Carcinogen

SECTION 16: OTHER INFORMATION

Prepared By: Health & Hygiene/ELB
420 Gallimore Dairy Road
Greensboro, NC 27409
(910) 665-1818

Date of Preparation: 9/17/97 ***Supercedes:***

MSDS Status: No revisions to date of printing.

The information herein is given in good faith, but no warranty, expressed or implied, is made.
Consult JMC (USA), Inc. for further information.

Appendix II. ADS ICP Results

Table A-II-1. ICP-MS Results on LANA85

ICP-MS Results **File Name:** Shanahan 3680,3690

Instrument: Agilent: Bldg 773A, Rm B142 **Analysis Date:** 10/25/2016

Analyst: Mark Jones **Reviewer Name:** Mark Jones

Comments: 100Kx IDF Al La Reported %RSD values reflect variance of replicate measurements.

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor Method Uncertanty is 20%

m/z	**Opening Check Standard** **ug/L**		**ADS Blank** **LW3680** **100 X** **ug/g**		**LaNi4.15Al0.85** **LW3690** **94.0911 X** **ug/g**		**Closing Check Standard** **ug/L**	
Al	1.03E+01	(9.36E-01 %RSD)	< 1.00E+01	(N/A %RSD)	5.95E+04	(3.31E+00 %RSD)	1.04E+01	(1.14E-01 %RSD)
V	9.70E+00	(4.33E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.94E+00	(5.11E-01 %RSD)
Co	1.00E+01	(1.44E+00 %RSD)	< 5.00E+00	(N/A %RSD)	7.24E+00	(1.09E+00 %RSD)	1.01E+01	(7.62E-01 %RSD)
Ga	1.03E+01	(1.29E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.04E+01	(1.27E+00 %RSD)
Rb	1.02E+01	(8.27E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.04E+01	(2.87E-01 %RSD)
Sr	1.02E+01	(8.32E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.03E+01	(3.86E-01 %RSD)
Y	1.01E+01	(1.52E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.03E+01	(7.37E-01 %RSD)
Zr	9.53E+00	(4.89E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.62E+00	(5.69E-02 %RSD)
Nb	1.01E+01	(1.40E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(6.01E-01 %RSD)
Mo	9.93E+00	(1.22E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(4.57E-01 %RSD)
Ru	9.97E+00	(1.12E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(1.62E+00 %RSD)
Rh	1.01E+01	(7.18E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.03E+01	(5.82E-01 %RSD)
Pd	1.00E+01	(6.05E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(3.16E-01 %RSD)
Ag	1.02E+01	(6.59E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.03E+01	(1.79E-01 %RSD)
Cd	9.95E+00	(9.82E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.02E+01	(1.51E+00 %RSD)
Sn	9.96E+00	(2.23E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.02E+01	(1.47E-01 %RSD)
Sb	1.01E+01	(1.26E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.02E+01	(1.45E-01 %RSD)
Te	1.01E+01	(1.50E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.03E+01	(7.20E-01 %RSD)
Cs	9.96E+00	(6.44E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(3.54E-01 %RSD)
Ba	9.97E+00	(3.13E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.02E+01	(1.16E+00 %RSD)
La	1.05E+01	(1.03E-01 %RSD)	< 5.00E+00	(N/A %RSD)	3.53E+05	(9.40E-01 %RSD)	1.04E+01	(4.05E-01 %RSD)
Ce	1.01E+01	(9.59E-01 %RSD)	< 1.00E+01	(N/A %RSD)	< 9.41E+00	(N/A %RSD)	1.03E+01	(1.16E+00 %RSD)
Pr	1.02E+01	(1.46E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.03E+01	(1.00E+00 %RSD)
Nd	1.01E+01	(1.55E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.03E+01	(1.05E+00 %RSD)
Sm	9.91E+00	(1.25E+00 %RSD)	< 5.00E+00	(N/A %RSD)	2.60E+01	(3.59E+00 %RSD)	1.00E+01	(4.29E-01 %RSD)
Eu	9.83E+00	(1.58E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.88E+00	(8.78E-01 %RSD)
Gd	9.95E+00	(1.72E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(7.46E-01 %RSD)
Tb	1.01E+01	(1.57E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(1.18E+00 %RSD)

Dy	9.75E+00	(1.17E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.77E+00	(6.28E-01 %RSD)
Ho	9.92E+00	(9.93E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.98E+00	(1.12E-01 %RSD)
Er	9.79E+00	(1.44E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.73E+00	(1.01E+00 %RSD)
Tm	1.00E+01	(1.07E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.01E+01	(1.36E-01 %RSD)
Yb	9.87E+00	(1.15E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.85E+00	(4.59E-02 %RSD)
Lu	1.02E+01	(9.96E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	1.02E+01	(1.87E-01 %RSD)
Hf	9.77E+00	(8.10E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.69E+00	(1.51E+00 %RSD)
Ta	9.93E+00	(4.87E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.94E+00	(8.24E-01 %RSD)
W	9.59E+00	(8.85E-01 %RSD)	< 5.00E+00	(N/A %RSD)	1.47E+01	(2.91E-01 %RSD)	9.51E+00	(1.21E+00 %RSD)
Re	9.69E+00	(8.32E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.59E+00	(1.16E+00 %RSD)
Ir	1.01E+01	(1.03E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.93E+00	(6.25E-01 %RSD)
Pt	9.77E+00	(4.91E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.72E+00	(1.21E-01 %RSD)
Tl	1.01E+01	(5.77E-01 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.96E+00	(6.02E-02 %RSD)
Pb	1.00E+01	(1.37E+00 %RSD)	< 5.00E+00	(N/A %RSD)	< 4.70E+00	(N/A %RSD)	9.83E+00	(1.73E-01 %RSD)

Table A-II-2. ICP-MS Results on LANA75

ICP-MS Results

File Name:	Shanahan 1695,1696	**Analysis Date: 6/27/2016**	
Instrument:	Agilent: Bldg 773A, Rm B142	**Reviewer Name: Mark Jones**	
Analyst:	Steve Moody		
Comments:	N/A		

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor

Method Uncertanty is +/- 20% Reported %RSD values reflect variance of replicate measurements.

			LW1695 ADS Blank		**LW1696 La-Ni-Al Dissolution of 1684**			
			90.9091 X		90.9918 X			
m/z	**Opening Check Standard** (ug/L)	--	ug/g	--	ug/g	--	**Closing Check Standard** (ug/L)	--
La	1.00E+01	(9.78E-01 %RSD)	< 4.55E+00	(N/A %RSD)	3.34E+05	(8.14E-01 %RSD)	1.01E+01	(1.95E+00 %RSD)
W	9.76E+00	(1.29E+00 %RSD)	< 4.55E+00	(N/A %RSD)	1.80E+01	(1.64E+00 %RSD)	9.90E+00	(9.52E-01 %RSD)
Sm	1.00E+01	(4.50E-01 %RSD)	< 4.55E+00	(N/A %RSD)	1.32E+01	(5.90E-01 %RSD)	1.01E+01	(1.60E+00 %RSD)
Co	1.02E+01	(8.61E-01 %RSD)	< 4.55E+00	(N/A %RSD)	5.07E+00	(1.15E+01 %RSD)	1.02E+01	(3.08E+00 %RSD)
Ce	1.01E+01	(2.13E-01 %RSD)	< 9.09E+00	(N/A %RSD)	< 9.10E+00		1.02E+01	(1.20E+00 %RSD)
V	9.92E+00	(9.56E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.83E+00	(1.46E+00 %RSD)
Ga	1.04E+01	(3.71E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.02E+01	(1.90E+00 %RSD)
Rb	1.01E+01	(8.14E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(8.46E-01 %RSD)
Sr	1.01E+01	(2.64E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.01E+01	(6.96E-01 %RSD)
Y	1.01E+01	(1.29E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.01E+01	(7.34E-01 %RSD)
Zr	9.89E+00	(1.62E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.81E+00	(1.03E+00 %RSD)
Nb	1.04E+01	(3.51E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.05E+01	(8.37E-01 %RSD)
Mo	9.92E+00	(7.81E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(1.97E+00 %RSD)
Ru	9.96E+00	(9.45E-02 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(3.03E-01 %RSD)
Rh	1.03E+01	(9.28E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.04E+01	(2.14E-01 %RSD)
Pd	1.01E+01	(1.06E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.01E+01	(8.57E-02 %RSD)
Ag	9.97E+00	(4.34E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.99E+00	(5.70E-01 %RSD)
Cd	1.01E+01	(8.00E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(9.68E-01 %RSD)
Sn	9.99E+00	(5.77E-02 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.98E+00	(5.41E-02 %RSD)
Sb	1.01E+01	(9.18E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(2.30E-01 %RSD)
Te	1.01E+01	(2.13E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(2.45E+00 %RSD)
Cs	9.97E+00	(8.55E-02 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(1.07E+00 %RSD)
Ba	1.02E+01	(1.31E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.03E+01	(9.31E-01 %RSD)
Pr	1.01E+01	(1.15E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.02E+01	(7.37E-01 %RSD)
Nd	1.00E+01	(4.93E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.00E+01	(1.69E+00 %RSD)
Eu	1.00E+01	(5.02E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.02E+01	(1.01E+00 %RSD)
Gd	9.92E+00	(3.21E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.01E+01	(1.09E+00 %RSD)
Tb	1.01E+01	(7.53E-02 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.02E+01	(1.77E-01 %RSD)
Dy	9.77E+00	(1.31E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.92E+00	(2.10E+00 %RSD)
Ho	1.00E+01	(1.86E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.03E+01	(1.20E+00 %RSD)
Er	9.74E+00	(5.53E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.86E+00	(1.64E+00 %RSD)

Tm	1.01E+01	(1.88E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.02E+01	(2.35E-03 %RSD)
Yb	9.87E+00	(1.94E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.93E+00	(4.70E-01 %RSD)
Lu	1.01E+01	(1.38E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.02E+01	(1.67E+00 %RSD)
Hf	9.46E+00	(3.28E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.50E+00	(3.31E+00 %RSD)
Ta	1.01E+01	(4.20E-02 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.01E+01	(1.34E+00 %RSD)
Re	9.61E+00	(1.90E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		9.93E+00	(3.96E+00 %RSD)
Ir	9.99E+00	(3.81E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.01E+01	(1.57E+00 %RSD)
Pt	9.88E+00	(1.07E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.01E+01	(9.64E-01 %RSD)
Tl	1.02E+01	(1.53E-01 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.03E+01	(8.53E-01 %RSD)
Pb	1.00E+01	(1.49E+00 %RSD)	< 4.55E+00	(N/A %RSD)	< 4.55E+00		1.03E+01	(1.33E+00 %RSD)

Table A-II-3. ICP-ES Results on “LANA75"

ICP-OES Results

Description: ADS Generated Blank

Travel Copy: LW-AD-PROJ-160602-3

Instrument: Perkin-Elmer Optima ICP-ES

Reviewer: John Young

Comments: Majors on 1kx, all else on 100x

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.

Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_DESCRIPTION:	ADS Generated Blank	Dissolution of 1684
USER_SAMPLEID:	ADS Generated Blank	La-Ni-Al Dissolution of 1684
SAMPLE_ID:	LW1695	LW1696
SAMPLE:	LW1695 100x ADS Gen Blk	LW1696 Cx LaNiAl Dis 1684

Element	**ug/g**	**%RSD**	**ug/g**	**%RSD**
Al	< 809	N/A	47900	10.1
La	< 181	N/A	305000	10
Ni	< 133	N/A	528000	10.3
Na	< 1000	N/A	< 1000	N/A
Ce	< 1130	N/A	< 1130	N/A
Co	< 120	N/A	< 120	N/A
Ba	< 15.6	N/A	< 15.6	N/A
Nb	< 156	N/A	< 156	N/A
V	< 158	N/A	< 1580	N/A
Re	< 194	N/A	< 195	N/A
Si	< 1950	N/A	< 1960	N/A
Pb	< 224	N/A	< 224	N/A
Cu	< 227	N/A	< 227	N/A
Mo	< 267	N/A	< 268	N/A
P	< 335	N/A	< 335	N/A
Mn	< 36.1	N/A	< 36.1	N/A
Fe	< 37.6	N/A	< 37.6	N/A
Sr	< 4.29	N/A	< 4.29	N/A
S	< 441	N/A	< 441	N/A
B	< 447	N/A	< 447	N/A
Ag	< 458	N/A	< 458	N/A
Cd	< 49	N/A	< 49.1	N/A
Nd	< 490	N/A	< 491	N/A
Ca	< 517	N/A	< 518	N/A
K	< 5440	N/A	< 5440	N/A
Zn	< 59.3	N/A	< 593	N/A
Sn	< 593	N/A	< 594	N/A
Cr	< 70.5	N/A	< 70.6	N/A
Mg	< 7.48	N/A	< 74.9	N/A
Ti	< 78.5	N/A	< 78.6	N/A
Zr	< 90.8	N/A	< 90.9	N/A
Li	< 91.5	N/A	< 91.6	N/A

Table A-II-4. ICP-ES Results on “LANA85"

ICP-OES Results

Description: samples
Travel Copy: LW-AD-PROJ-160914-2
Instrument: Leeman Prodigy ICP-ES
Reviewer: Rachel Deese
Comments: Al, La, Ni 100x; Fe confirmed on 3 emission lines, La and Ni over calibration but within demonstrated linear range

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS Generated Blank		LaNi4.15Al0.85	
SAMPLE_ID:	LW3680		LW3690	
UNITS:	mg/L		mg/L	
Element				
Ag	< 15.3	(N/A %RSD)	< 14.4	(N/A %RSD)
Al	< 39.8	(N/A %RSD)	59200	(10 %RSD)
B	< 48.2	(N/A %RSD)	< 45.4	(N/A %RSD)
Ba	< 1.16	(N/A %RSD)	< 1.09	(N/A %RSD)
Be	< 0.483	(N/A %RSD)	< 0.454	(N/A %RSD)
Ca	< 12.4	(N/A %RSD)	< 11.6	(N/A %RSD)
Cd	< 16.1	(N/A %RSD)	< 15.1	(N/A %RSD)
Ce	< 40.1	(N/A %RSD)	< 37.7	(N/A %RSD)
Co	< 32.6	(N/A %RSD)	< 30.7	(N/A %RSD)
Cr	< 15.7	(N/A %RSD)	< 14.8	(N/A %RSD)
Cu	< 54.6	(N/A %RSD)	< 51.4	(N/A %RSD)
Fe	35.7	(20.4 %RSD)	< 22.3	(N/A %RSD)
Gd	< 35.2	(N/A %RSD)	< 33.1	(N/A %RSD)
K	< 294	(N/A %RSD)	< 276	(N/A %RSD)
La	< 12.3	(N/A %RSD)	348000	(10 %RSD)
Li	< 36.7	(N/A %RSD)	224	(10.2 %RSD)
Mg	< 2.38	(N/A %RSD)	28.8	(11.1 %RSD)
Mn	< 2.82	(N/A %RSD)	< 2.65	(N/A %RSD)
Mo	< 46.6	(N/A %RSD)	< 43.8	(N/A %RSD)
Na	< 50.8	(N/A %RSD)	< 47.8	(N/A %RSD)
Ni	< 105	(N/A %RSD)	591000	(10 %RSD)
P	< 208	(N/A %RSD)	< 196	(N/A %RSD)
Pb	< 456	(N/A %RSD)	< 429	(N/A %RSD)
S	< 13100	(N/A %RSD)	< 12300	(N/A %RSD)
Sb	< 235	(N/A %RSD)	< 221	(N/A %RSD)
Si	< 200	(N/A %RSD)	< 188	(N/A %RSD)
Sn	< 131	(N/A %RSD)	< 123	(N/A %RSD)
Sr	< 1.06	(N/A %RSD)	< 0.997	(N/A %RSD)
Th	< 99.3	(N/A %RSD)	< 93.4	(N/A %RSD)
Ti	< 89.6	(N/A %RSD)	< 84.3	(N/A %RSD)
U	< 515	(N/A %RSD)	< 485	(N/A %RSD)
V	< 6.09	(N/A %RSD)	< 5.73	(N/A %RSD)
Zn	< 22.5	(N/A %RSD)	< 21.2	(N/A %RSD)
Zr	< 7.07	(N/A %RSD)	< 6.65	(N/A %RSD)

Appendix III. SEM results

ADS conducted standard SEM/EDX studies on the virgin LANA75 and 85 materials. Figures A-III-1 through 6 are from LANA75, while Figures A-III-7 through –20 are from LANA85.

Figure A-III-1. LANA75, very low magnification (59X)

Figure A-III-2. LANA75, very low magnification (59X)

Figure A-III-3. LANA75, very low magnification (122X)

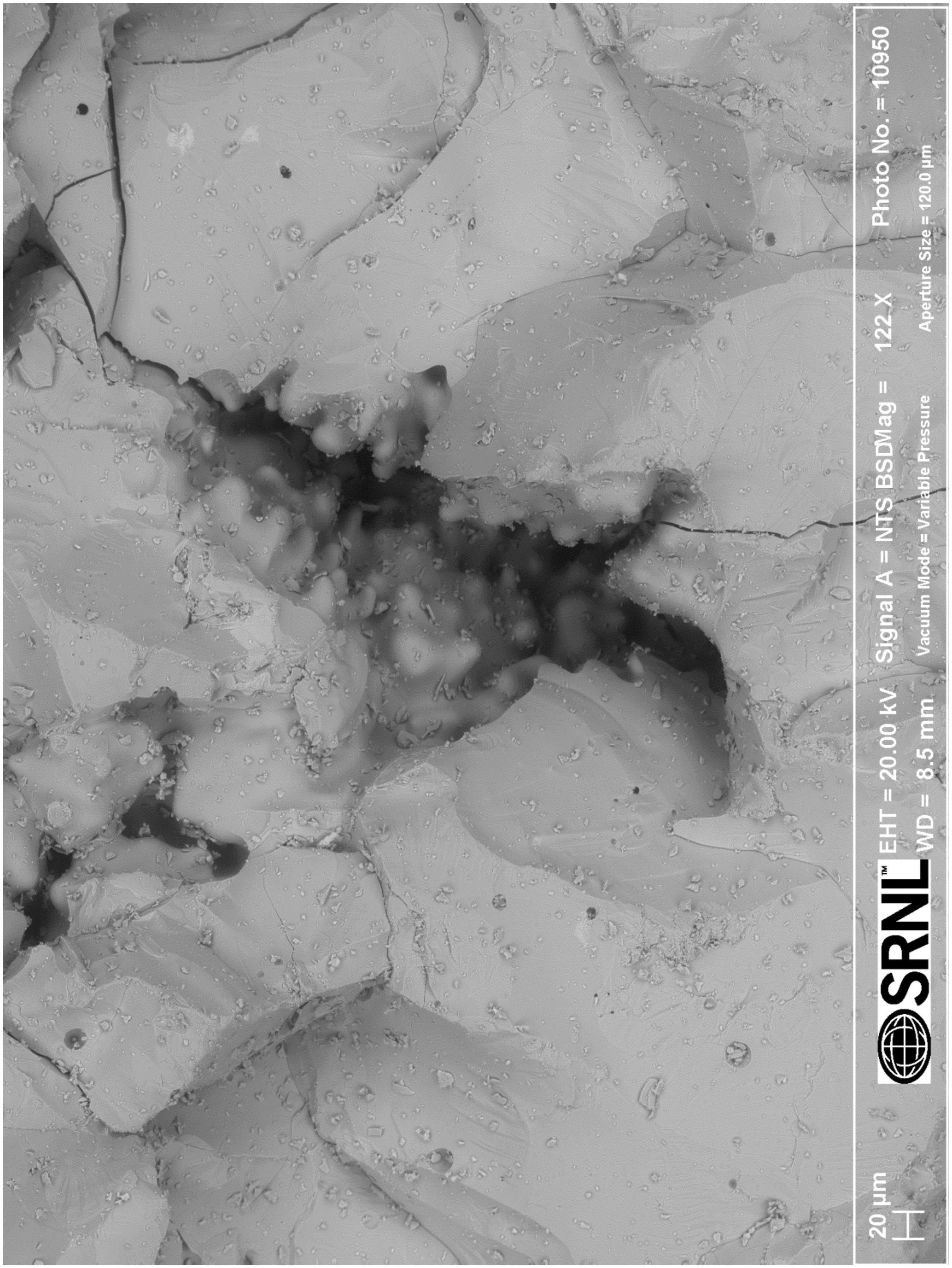


Figure A-III-4. LANA75, very low magnification (122X)

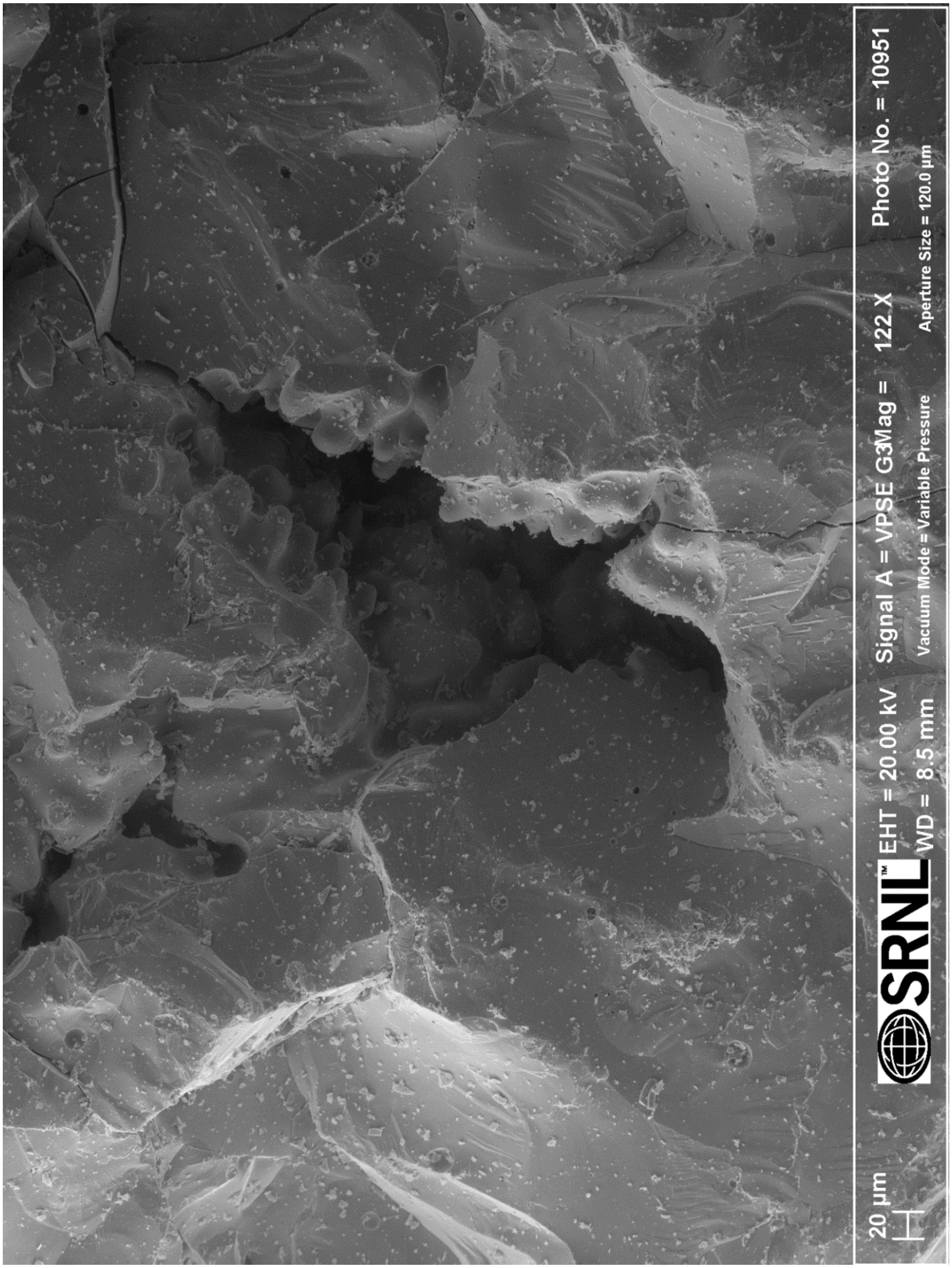


Figure A-III-5. LANA75, very low magnification (106X)

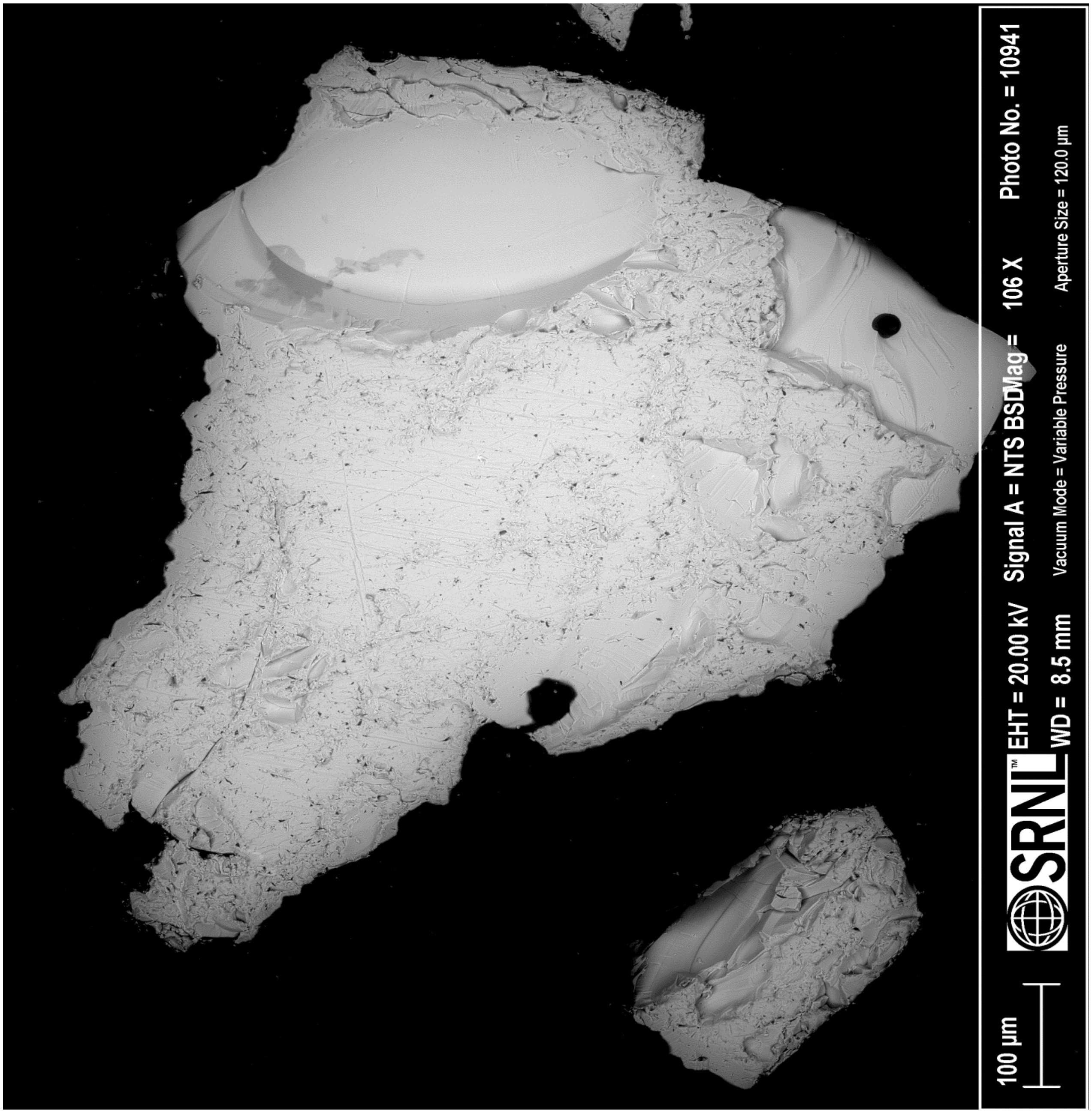


Figure A-III-6. LANA75, low magnification (263X)

Figure A-III-7. LANA85, very low magnification (12X)

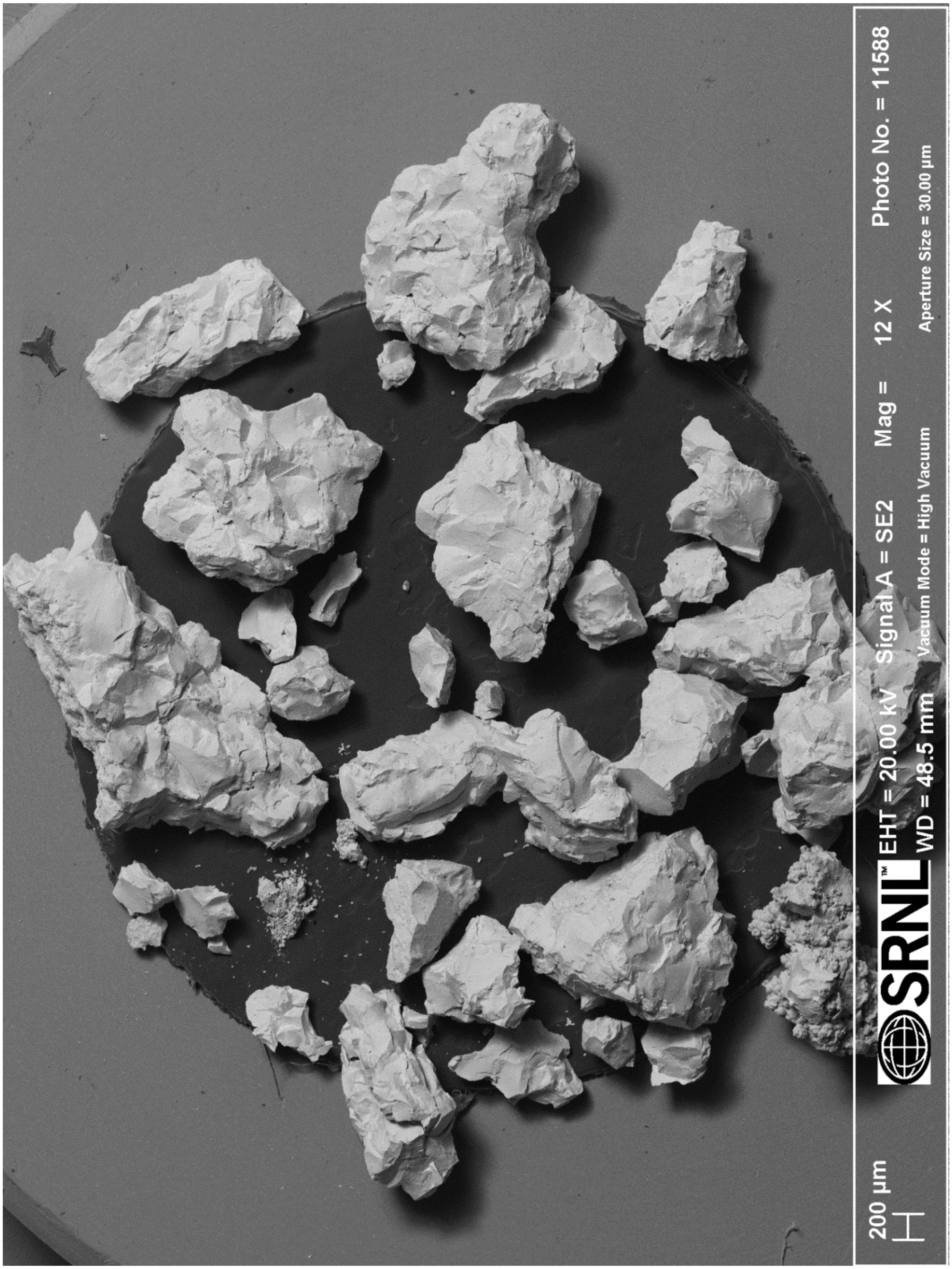


Figure A-III-8. LANA85, very low magnification (12X), box shows location where photo no. 11593 was taken (lower right corner)

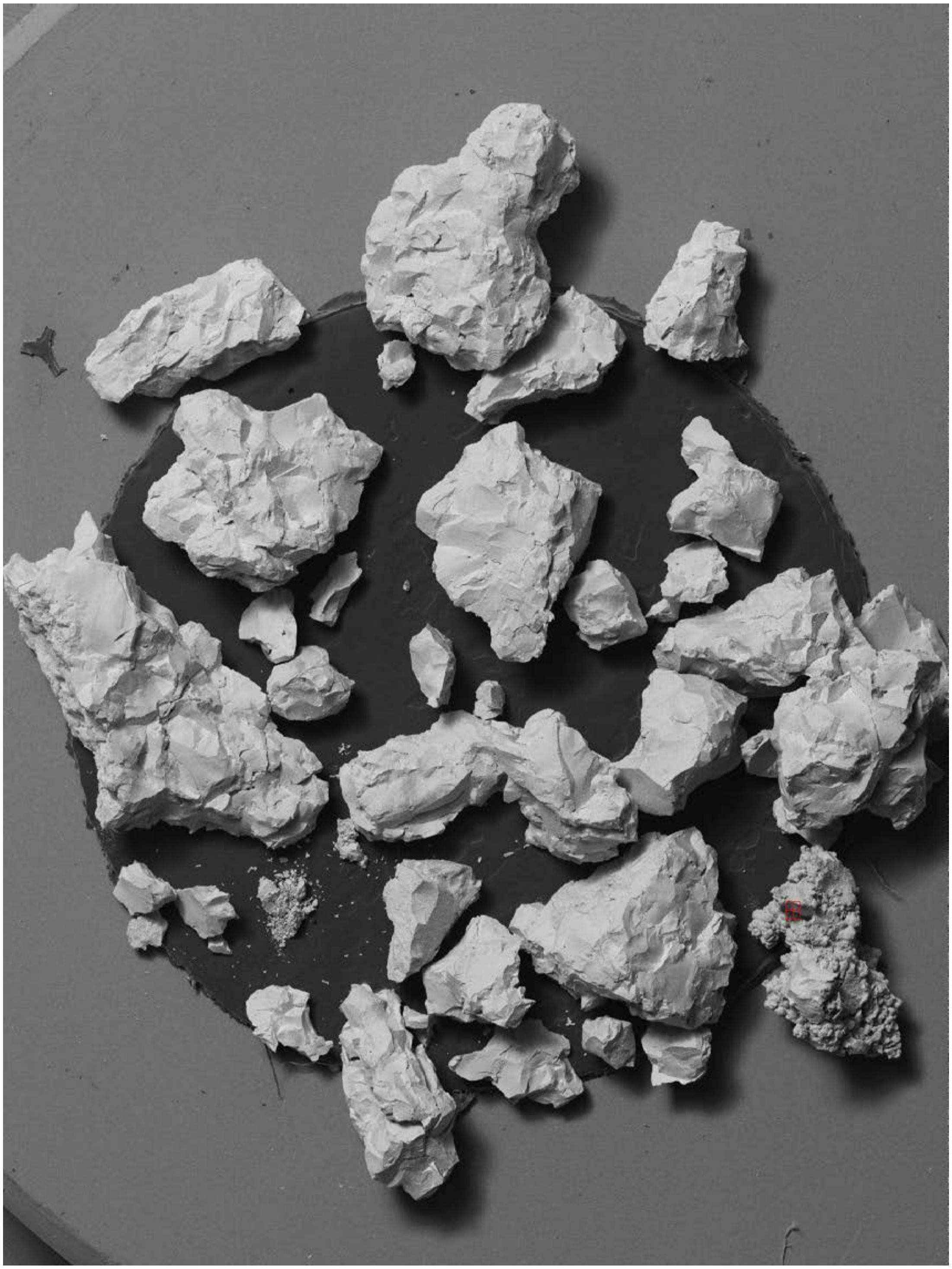


Figure A-III-9. LANA85, very low magnification (12X), box shows location where photo no. 11595 was taken (center of image, slightly to left)

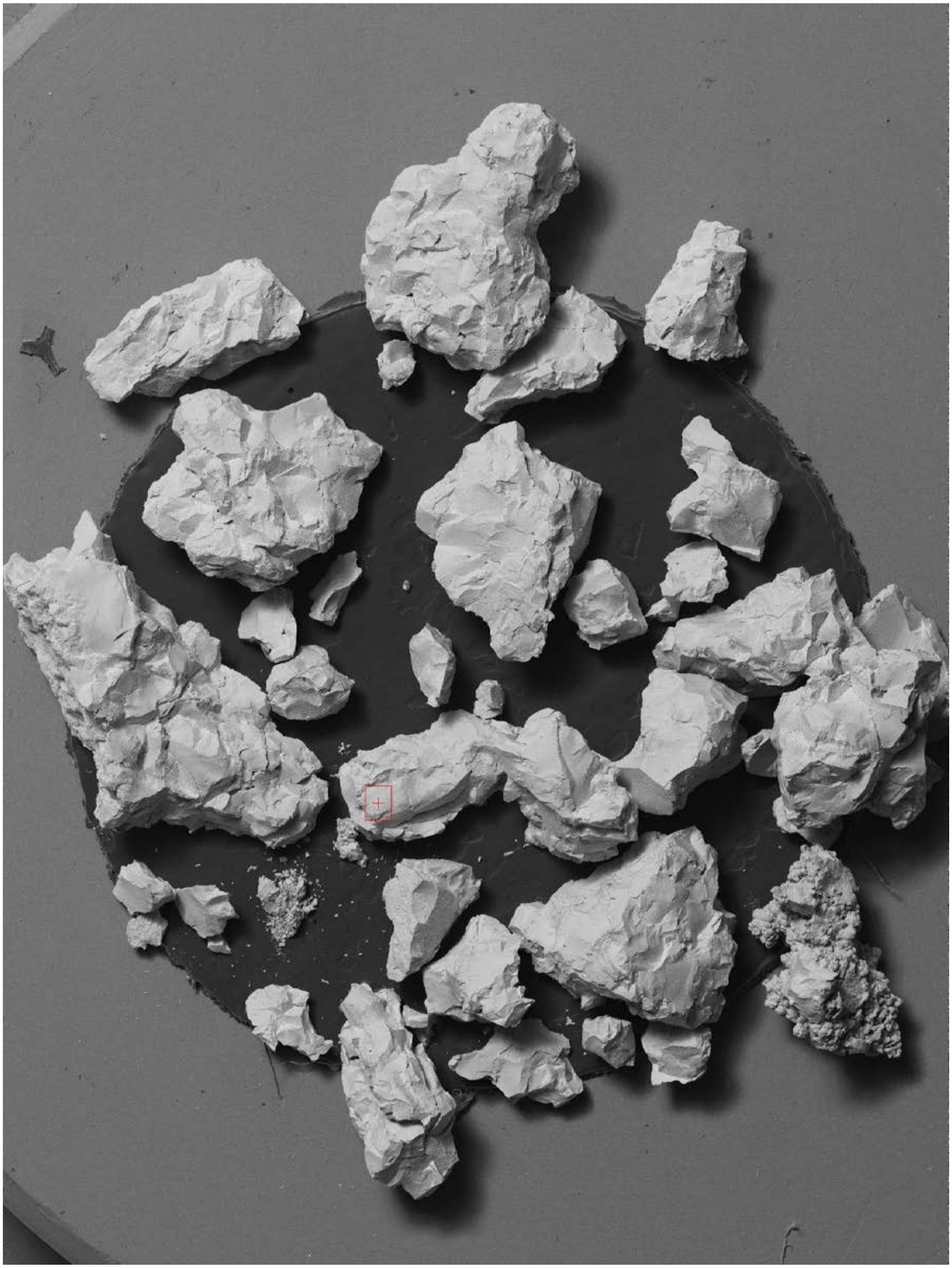


Figure A-III-10. LANA85, very low magnification (12X), box shows location where photo no. 11597 was taken (above center, large area)

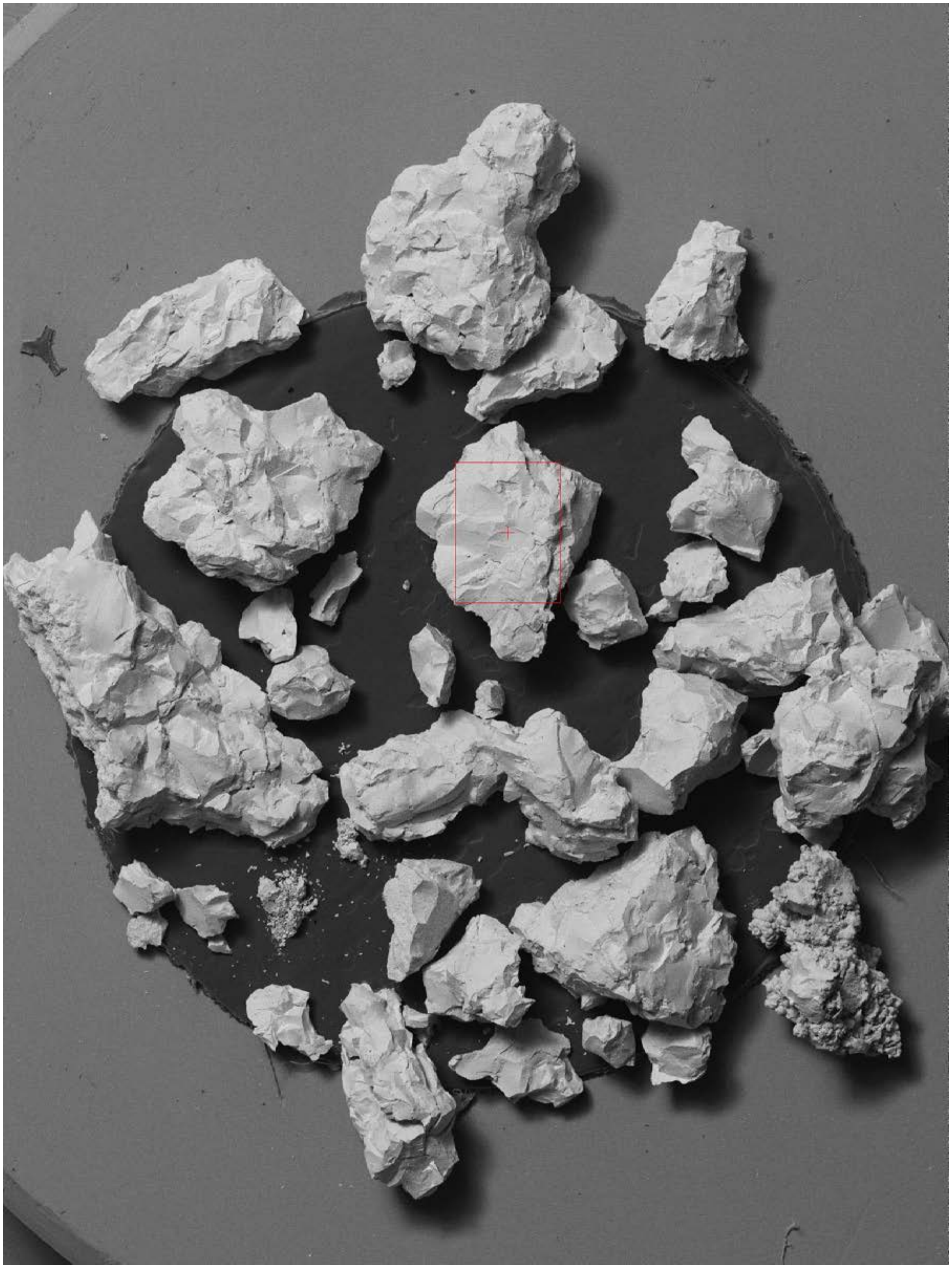


Figure A-III-11. LANA85, low mag (108X)

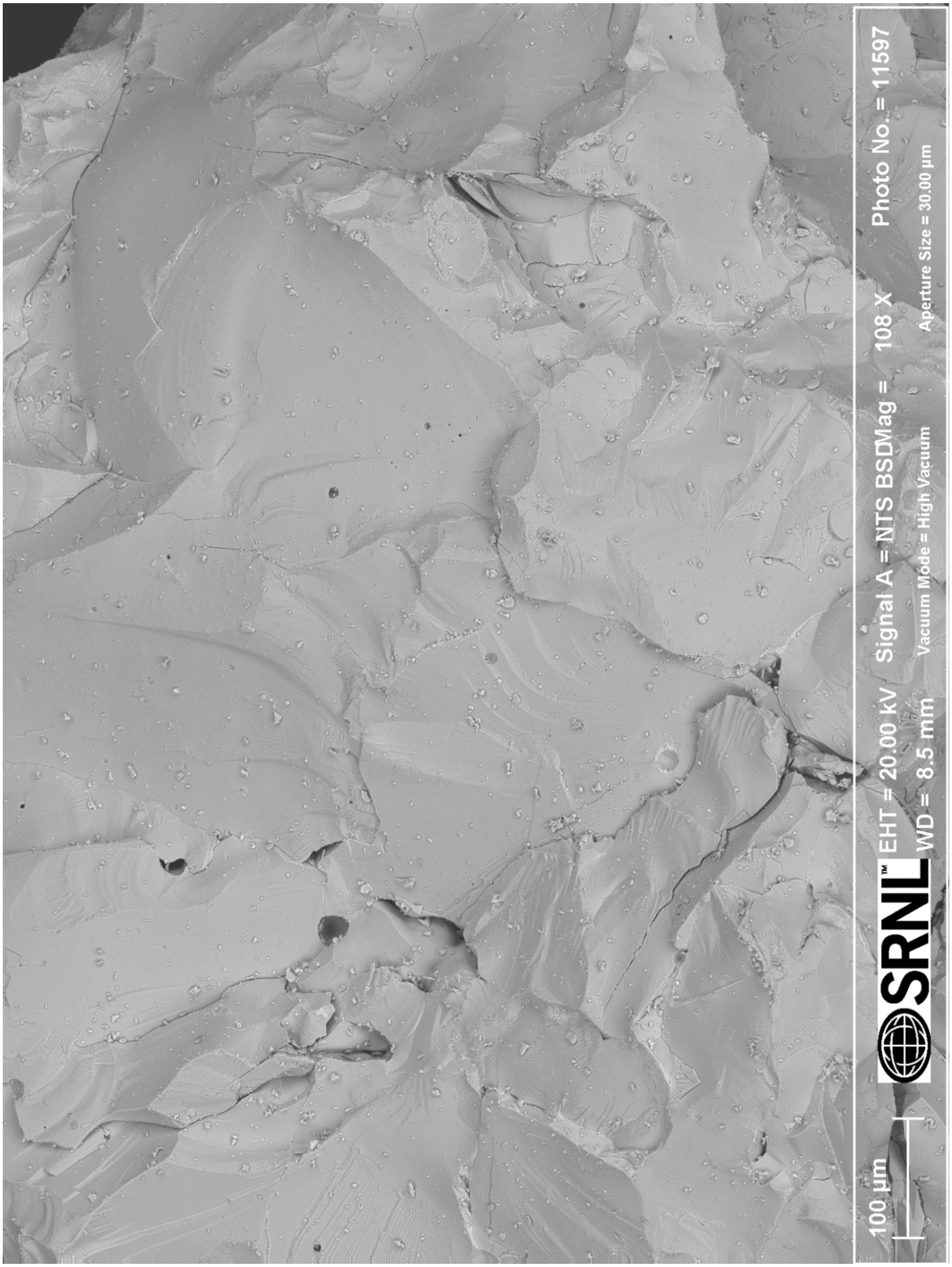


Figure A-III-12. LANA85, low mag (108X)

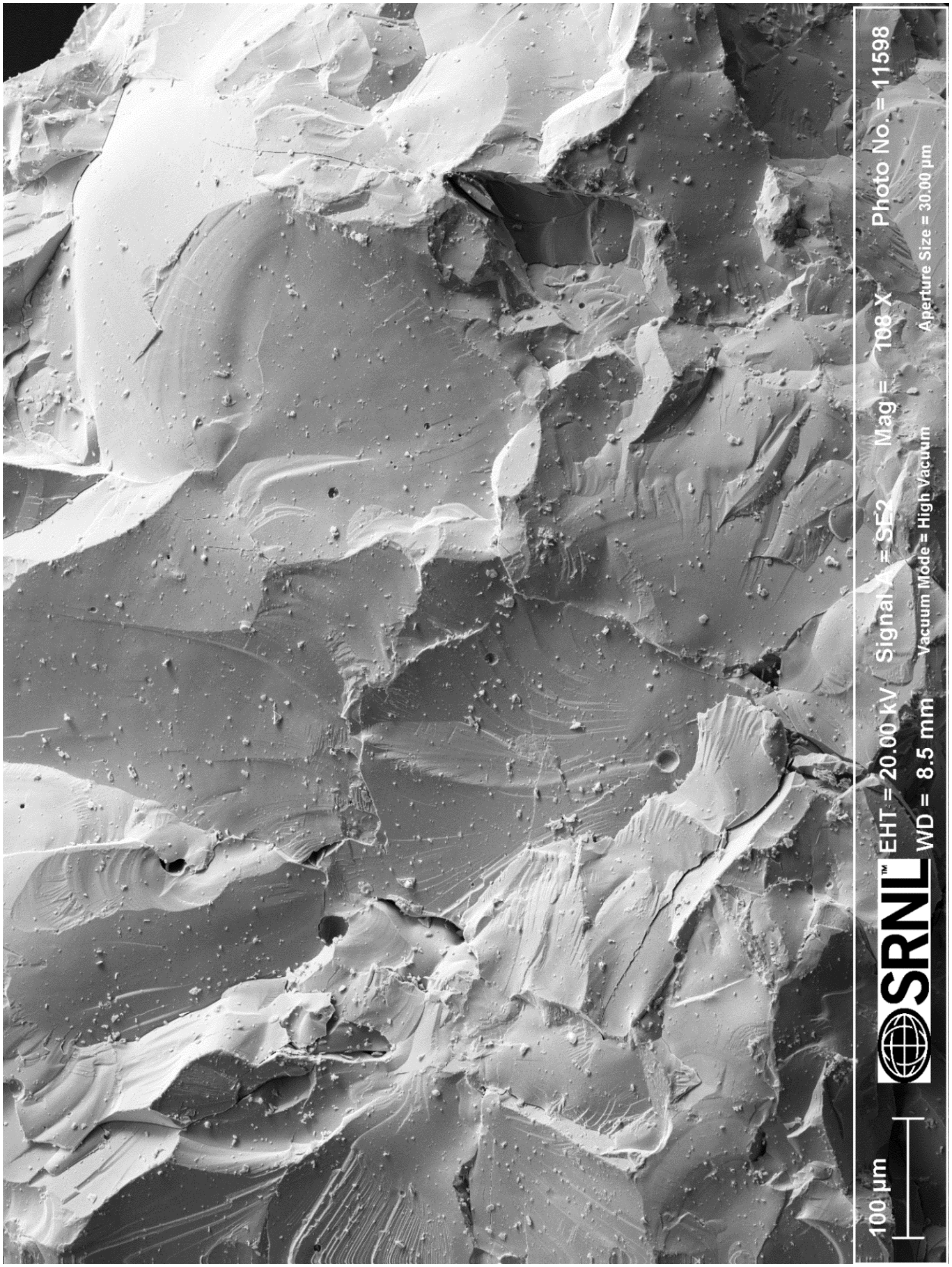


Figure A-III-13. LANA85, low mag (308X)

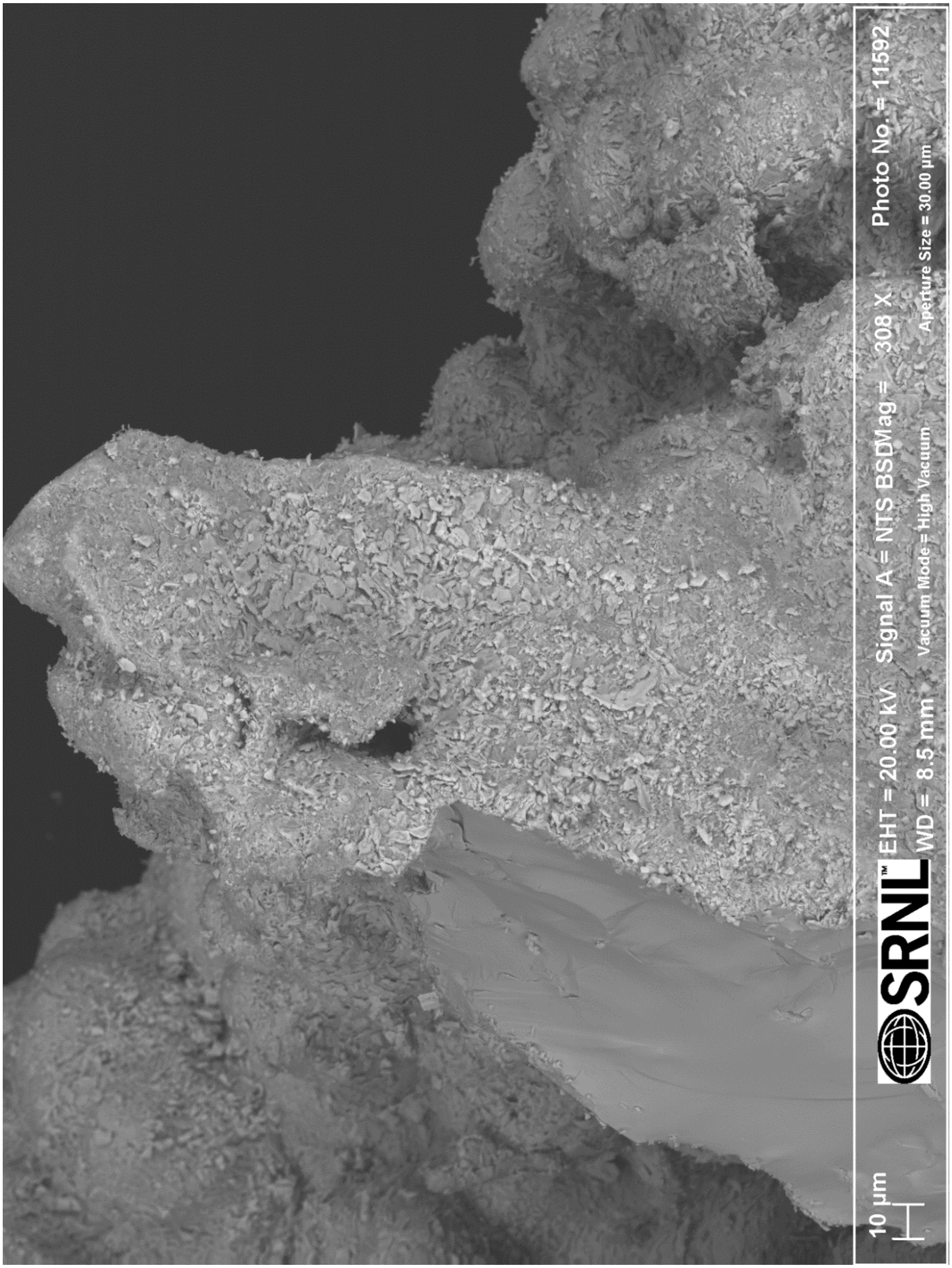


Figure A-III-14. LANA85, low mag (308X)

Figure A-III-15. LANA85, intermediate mag (442X)

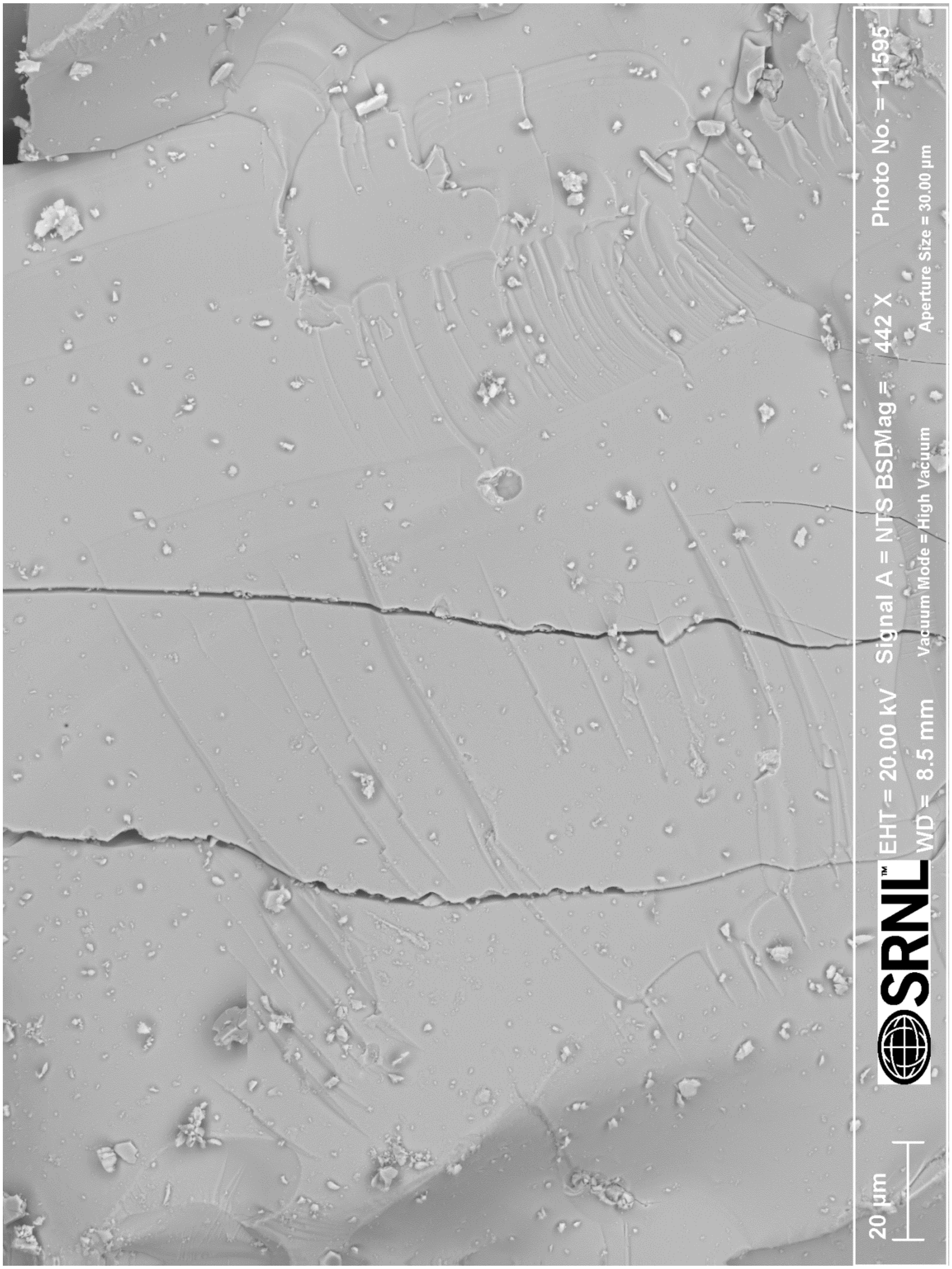


Figure A-III-16. LANA85, intermediate mag (442X)

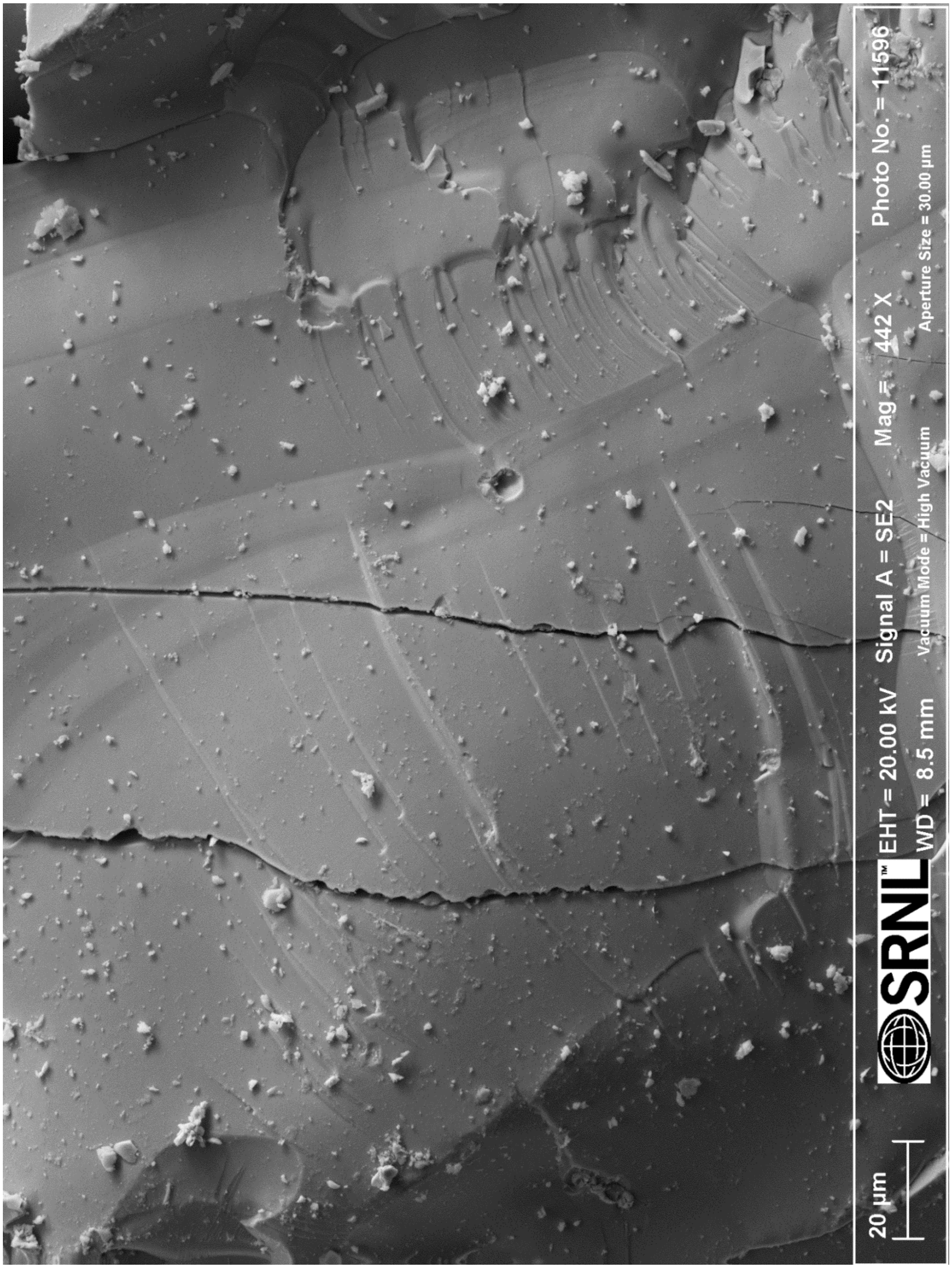


Figure A-III-17. LANA85, intermediate mag (852X)

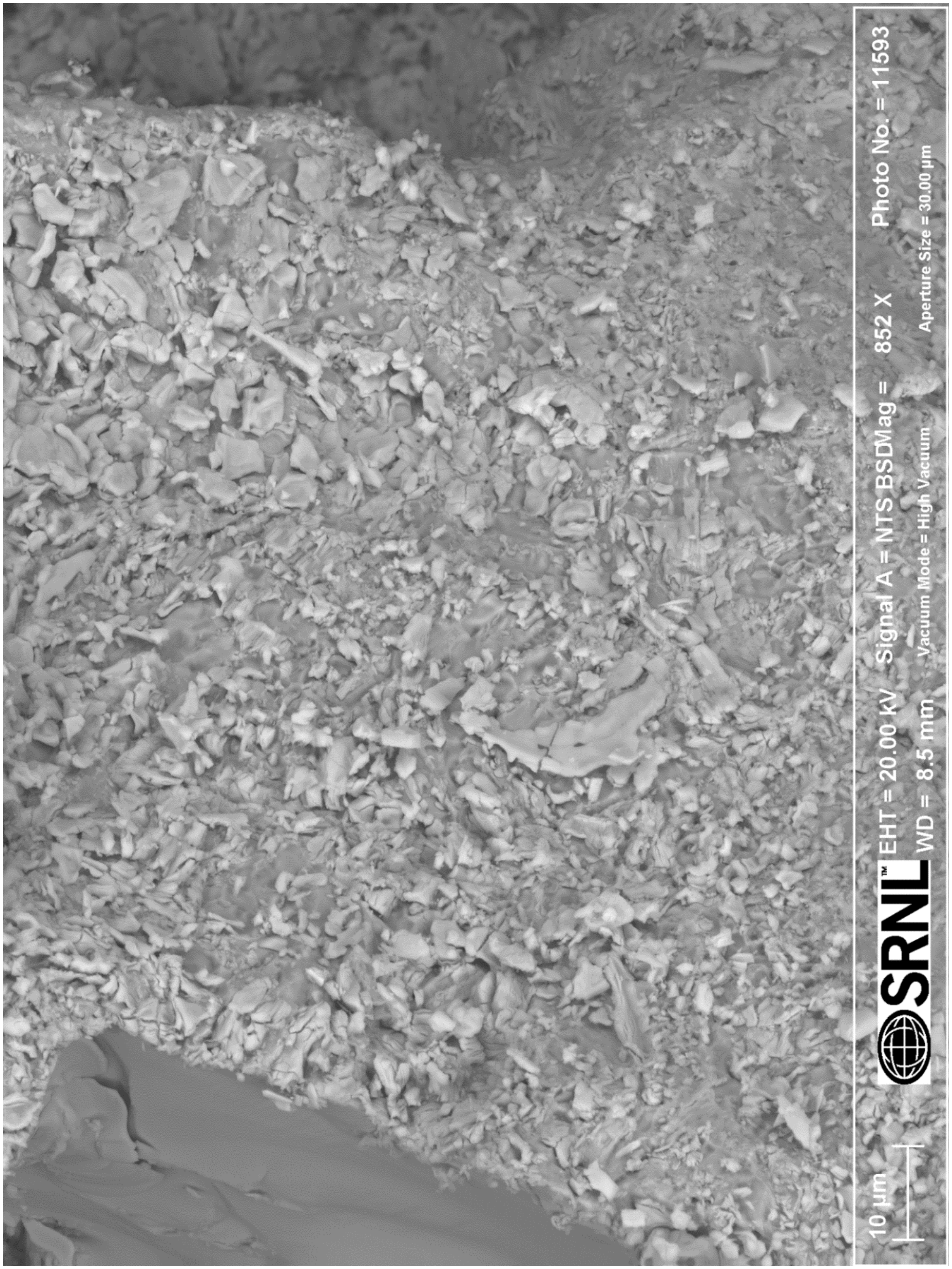


Figure A-III-18. LANA85, high mag (852X)

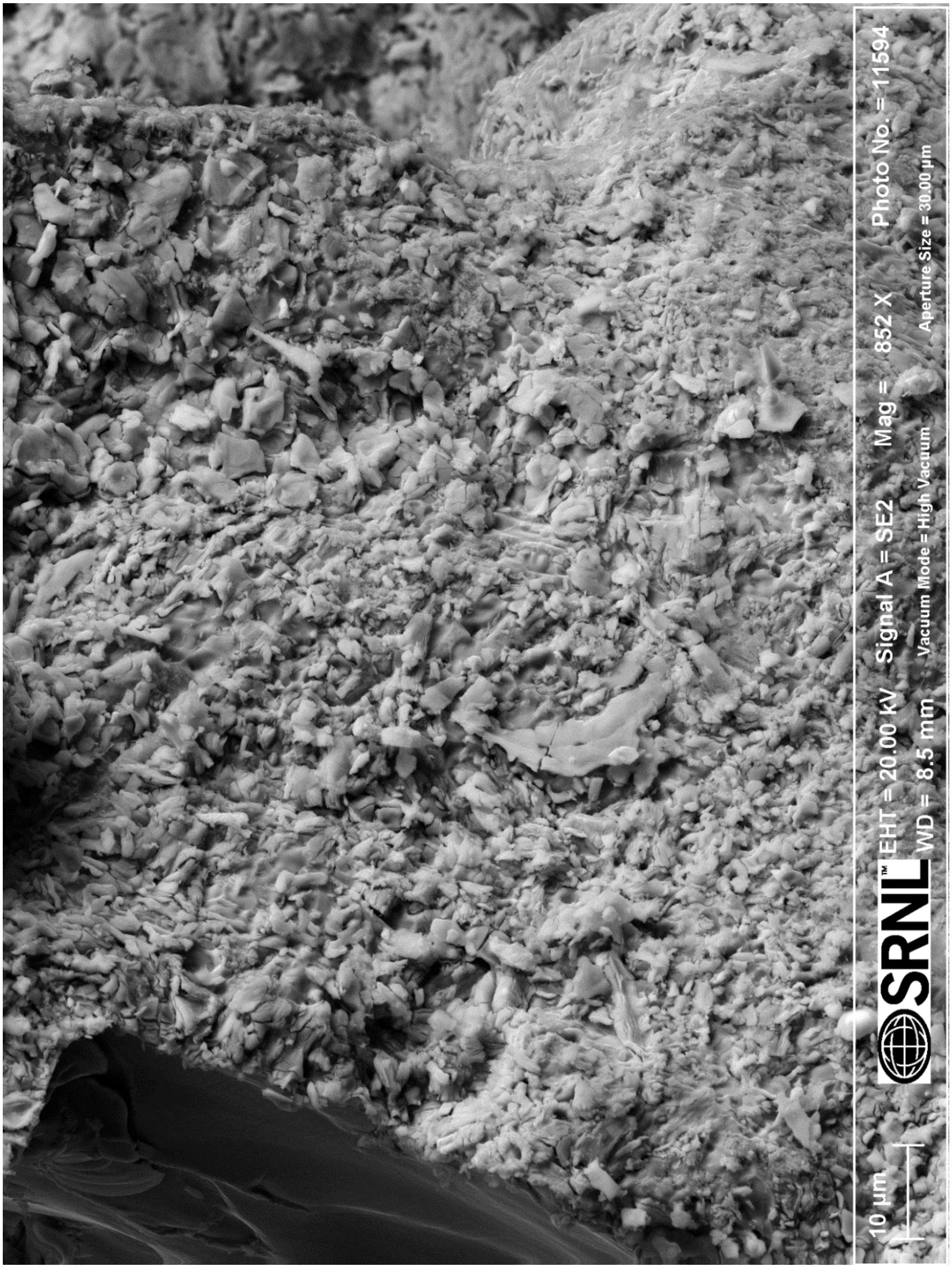


Figure A-III-19. LANA85, high mag (2000X)

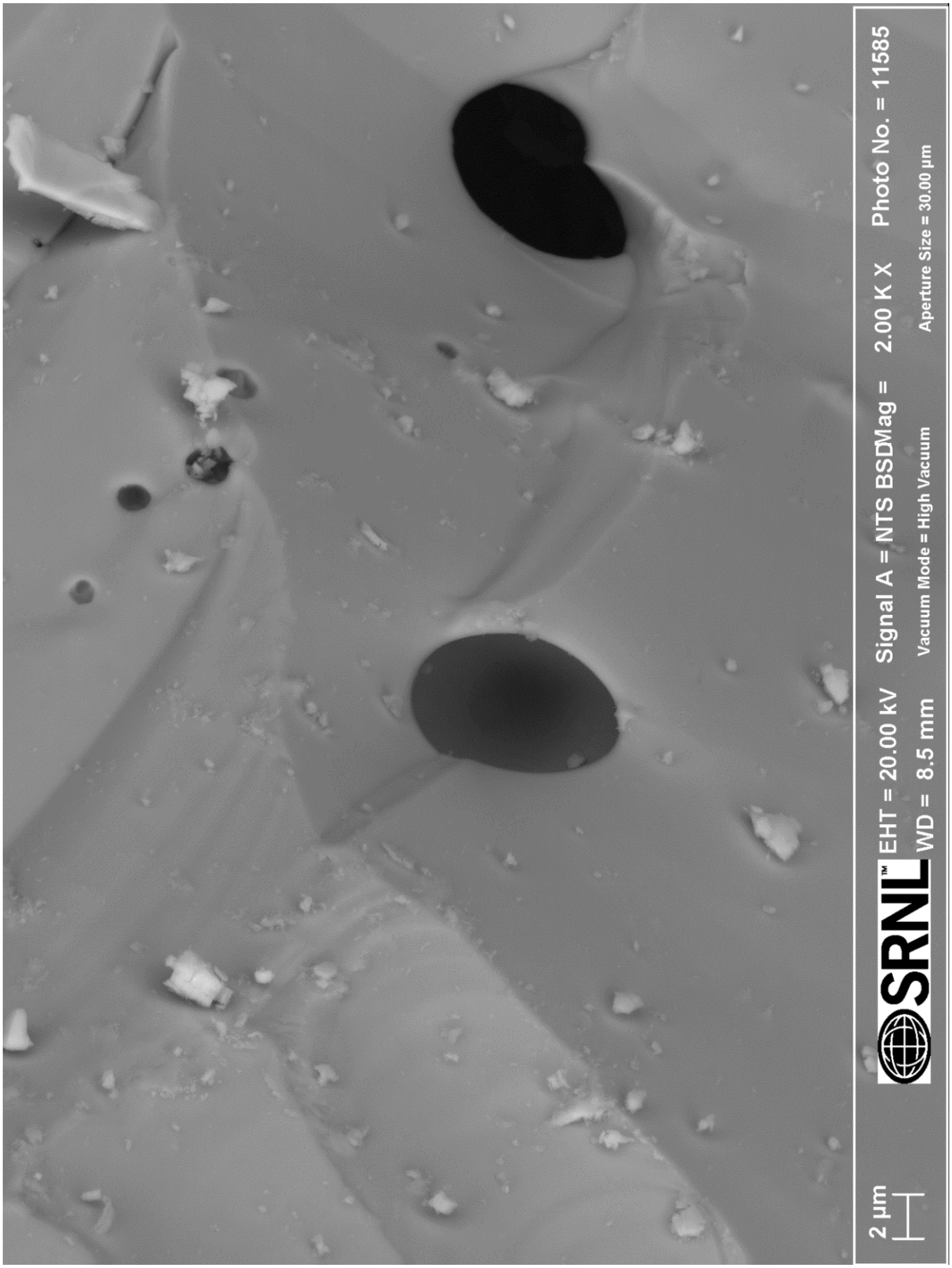
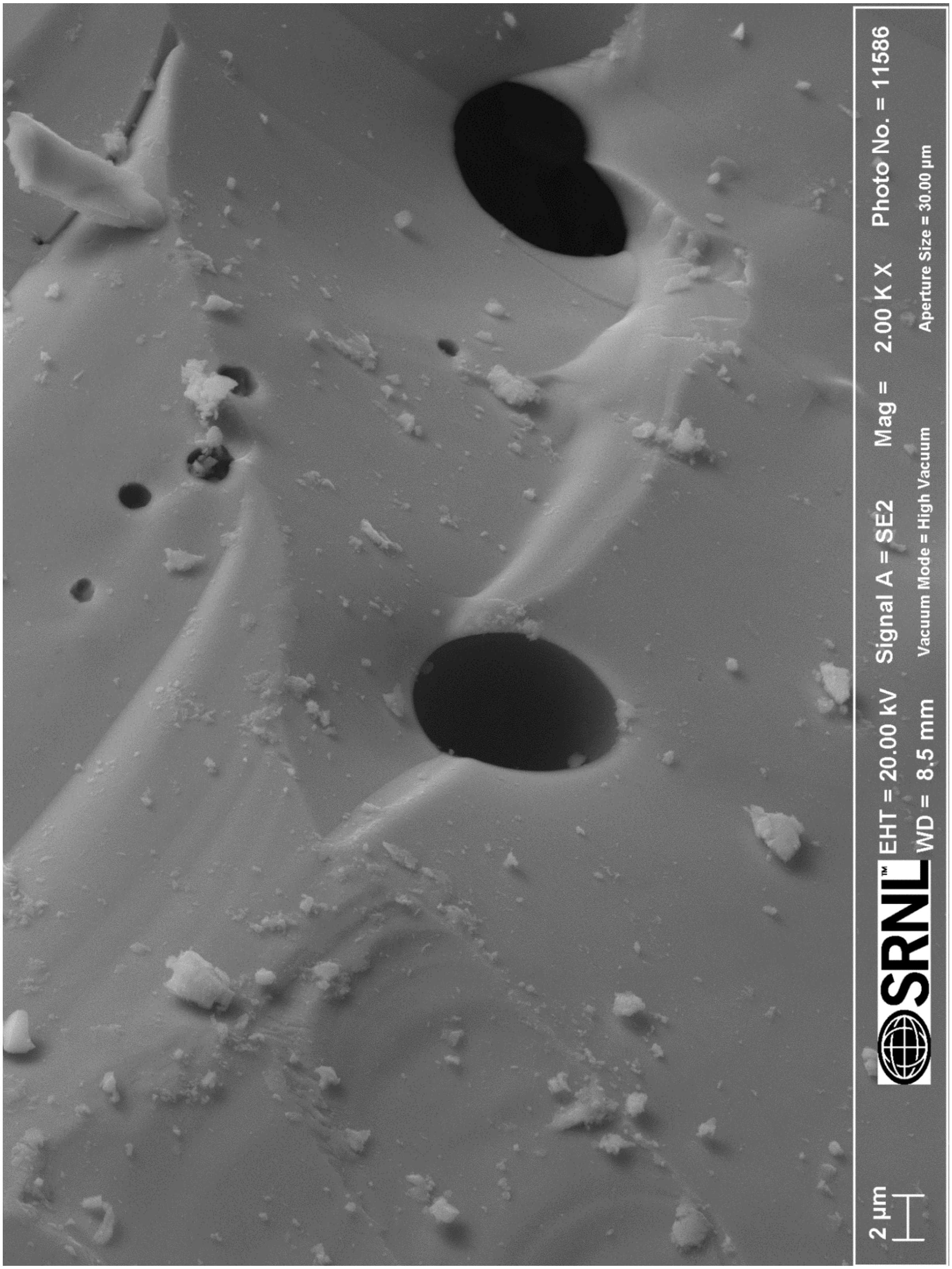


Figure A-III-20. LANA85, high mag (2000X)

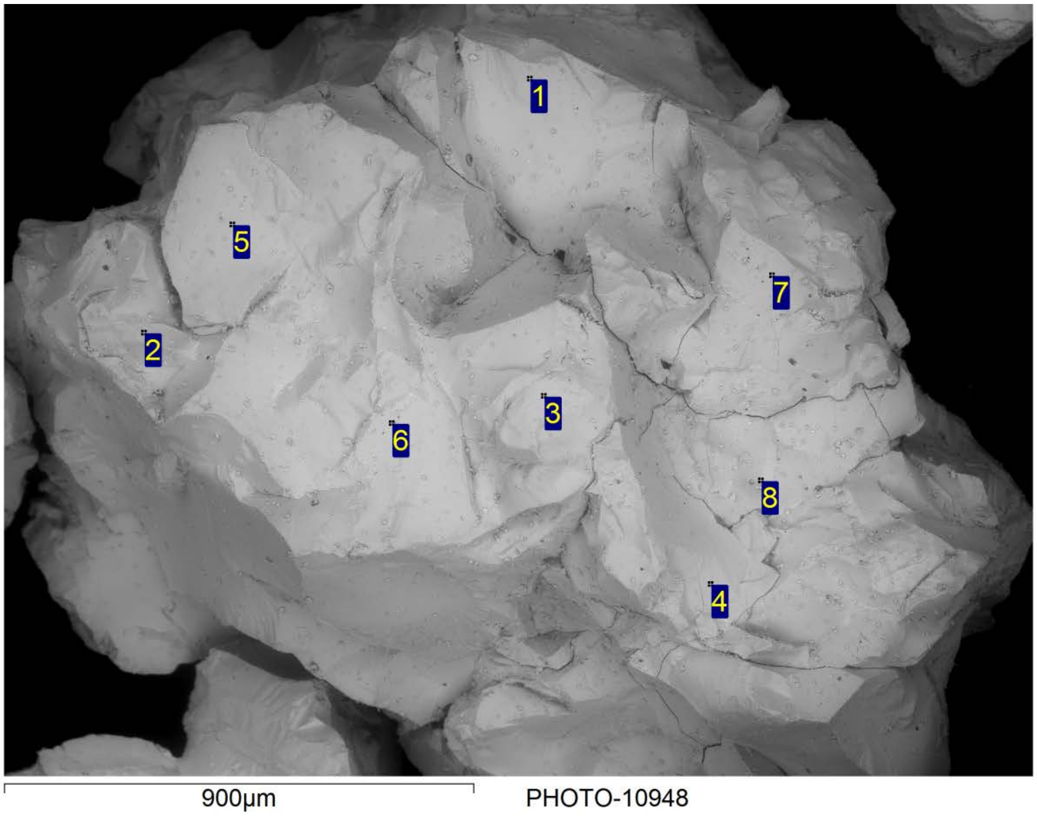


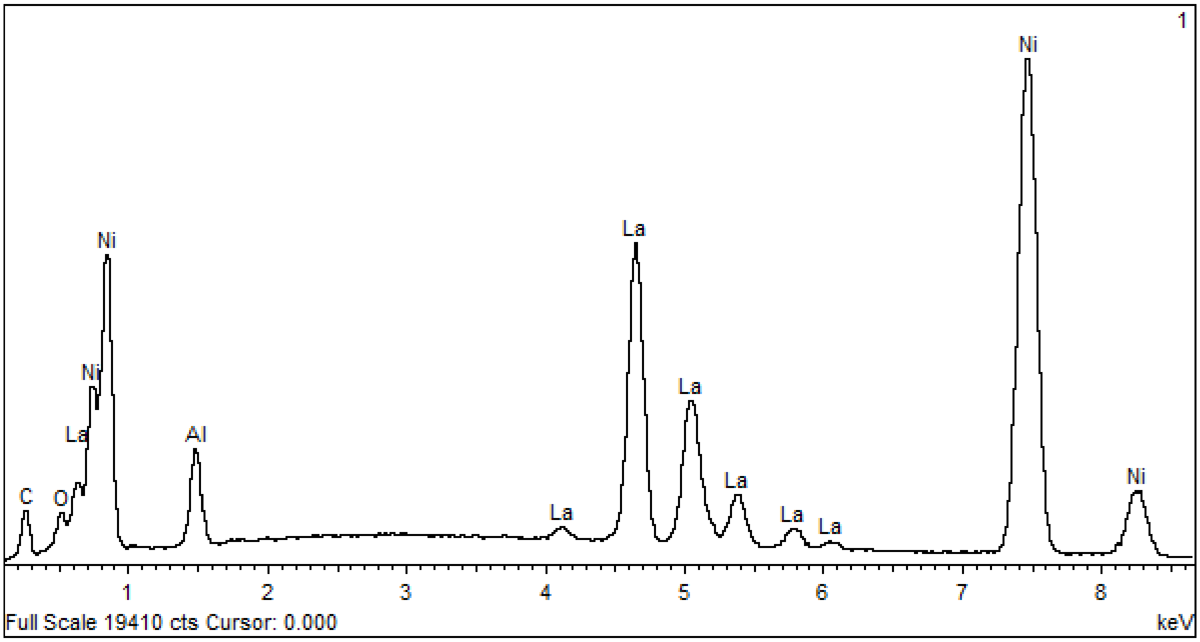
Appendix IV. ADS EDX results on LANA

The following two sections (Parts 1 and 2) are the written report from the ADS analyst. The numbers "1684" and "3683" are internal ADS LIMS numbers. Samples were examined as whole, unaltered particles and after cross-sectioning.

Part 1. LANA75 Material - LANA 1684 Whole particles (LANA75) Report from H. Ajo, ADS

Energy dispersive spectroscopy (EDS) and scanning electron microscope (SEM) imaging were performed on a LANA samples mounted on carbon sticky tape on an aluminum SEM stub, without carbon coating. SEM imaging and EDS conditions were 20 kV, variable pressure = 66 Pa nitrogen. Because the EDS was not performed at high vacuum, there is some overlap in spectra between regions due to the scattering of the electron beam by nitrogen. There is carbon and oxygen in the EDS spectra, due to the diffuse nature of the x-ray generating region, which may allow carbon from the epoxy to be detected, as well as the "adventitious" carbon film seen on most items not processed and analyzed in ultrahigh vacuum. The oxygen seen in all spectra may be due to residual oxygen in the SEM chamber or may be part of that "adventitious" carbon film.

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La
La
La
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La
Ni
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C
O
Ni
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2
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5
6
7
8
keV
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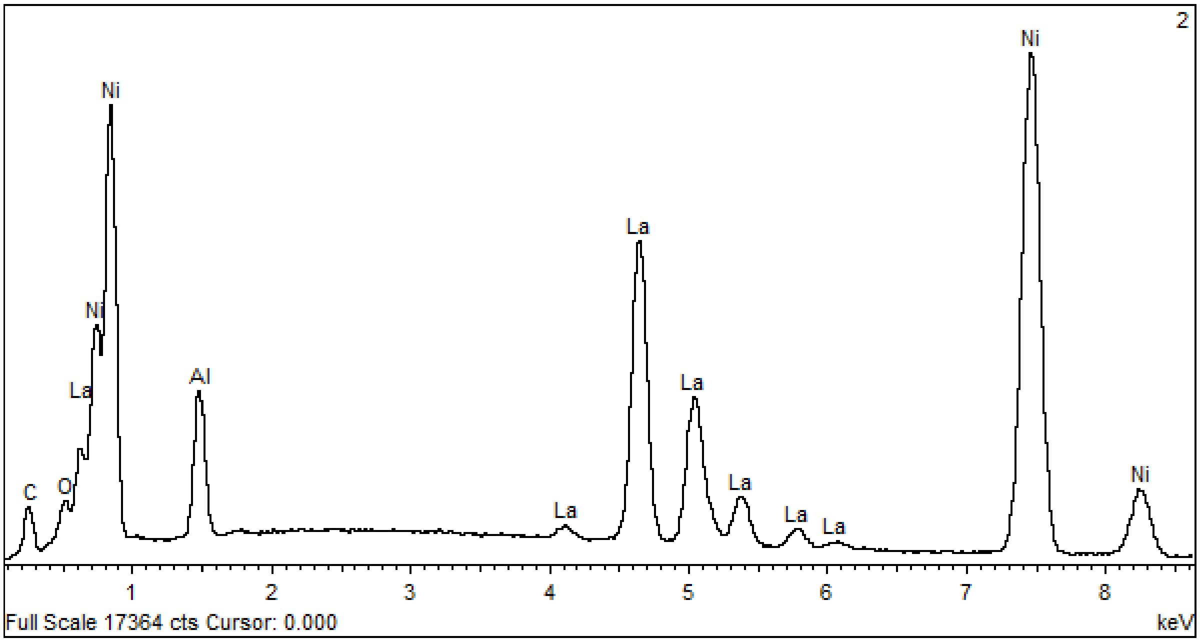
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Ni
La
La
La
La
La
La
Ni
Ni
La
Al
C
O
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3
4
5
6
7
8
keV
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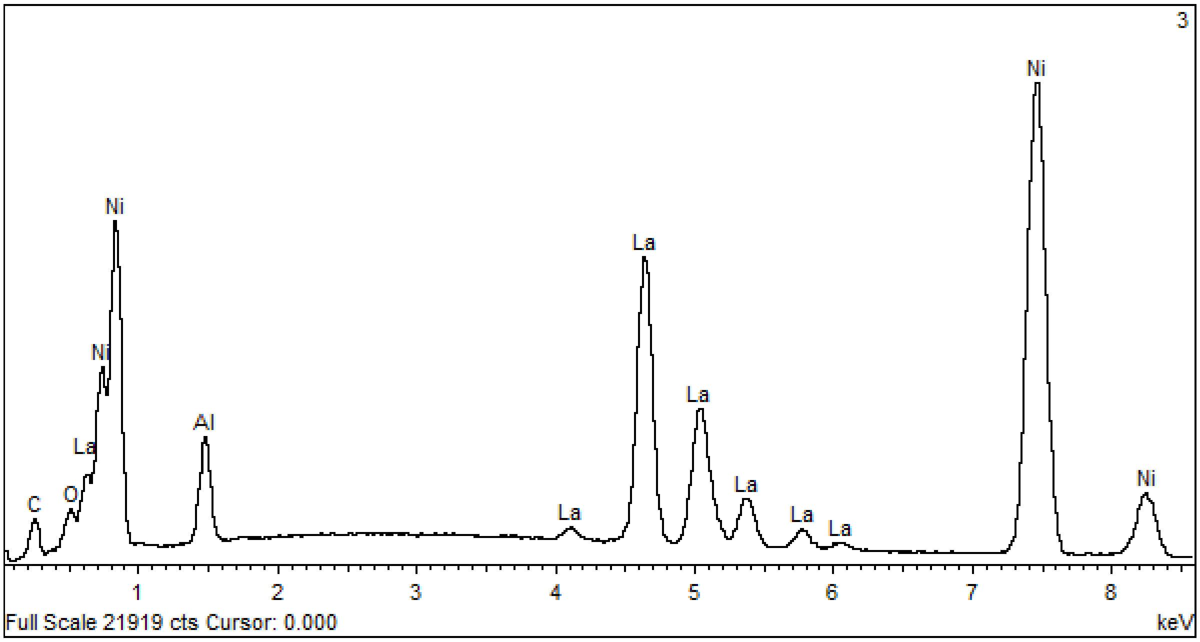
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Ni
La
C
O
Ni
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La
La
La
La
La
La
La
Ni
Ni
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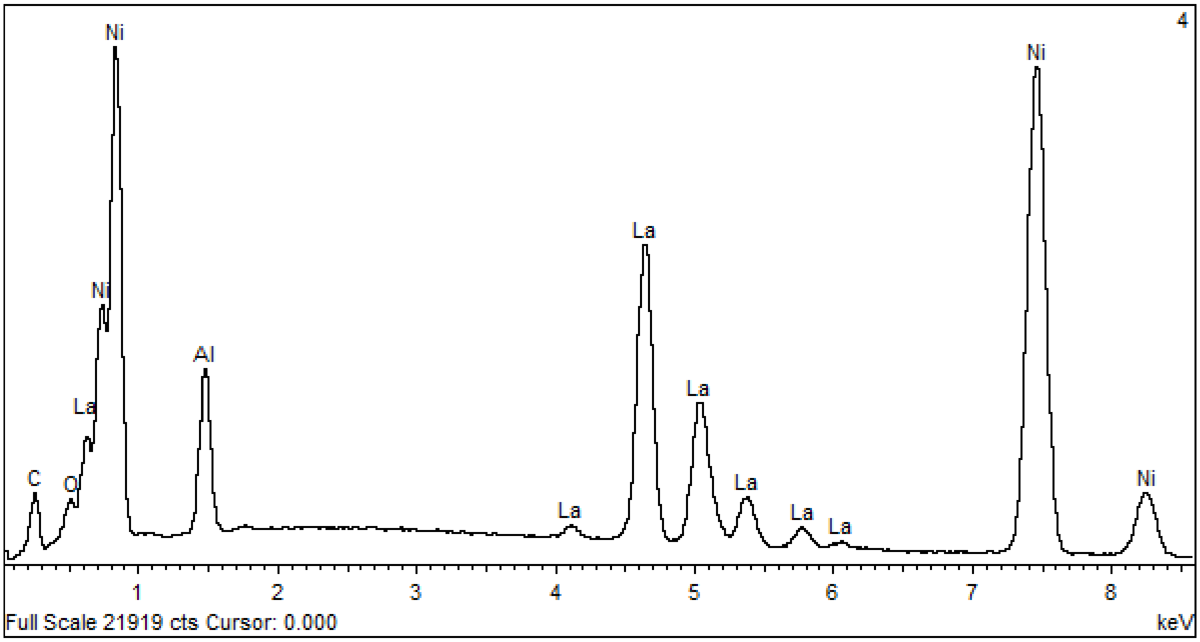
keV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Ni
La
C
O
Al
La
La
La
La
La
La
Ni
Ni
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3
4
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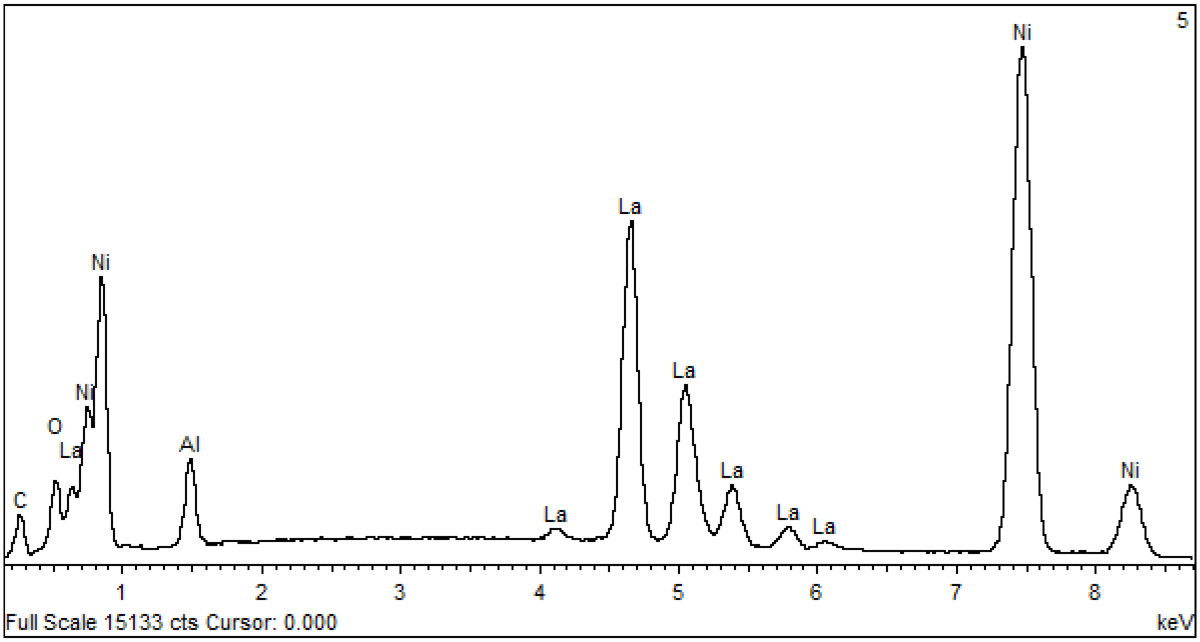
Full Scale 21919 cts Cursor: 0.000
keV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La
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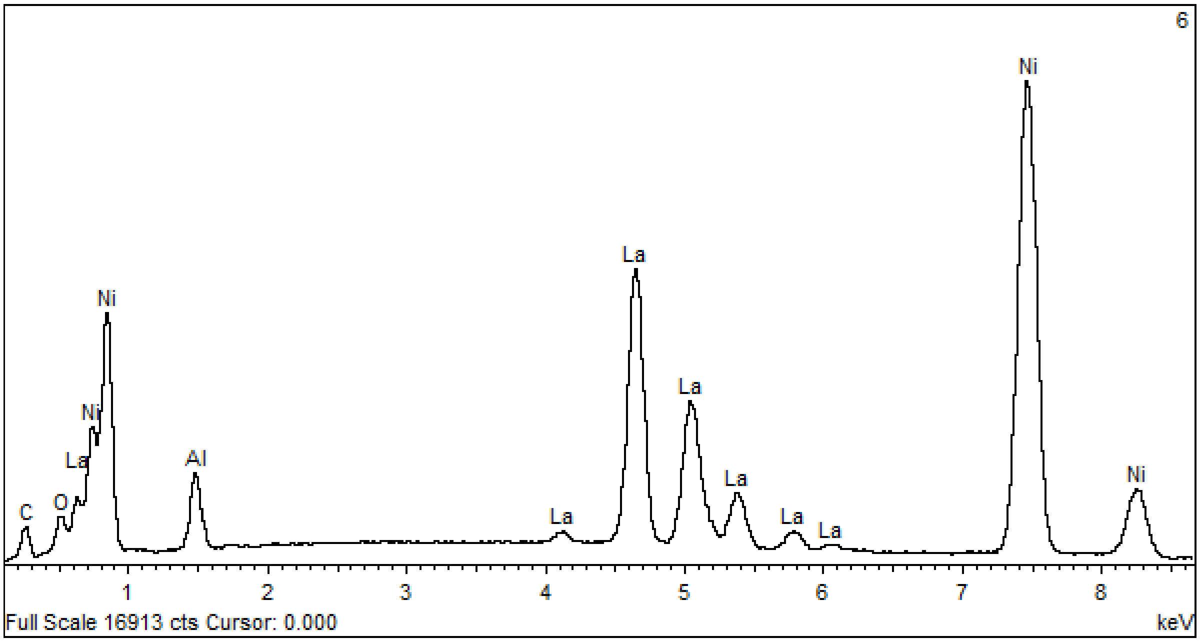
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La
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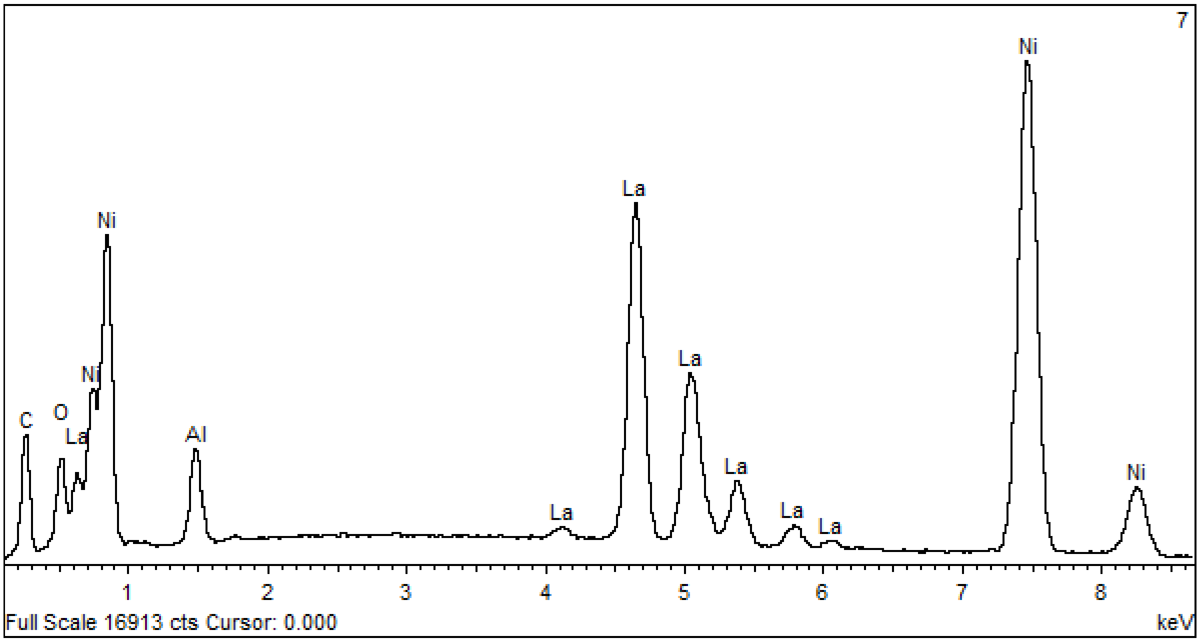
keV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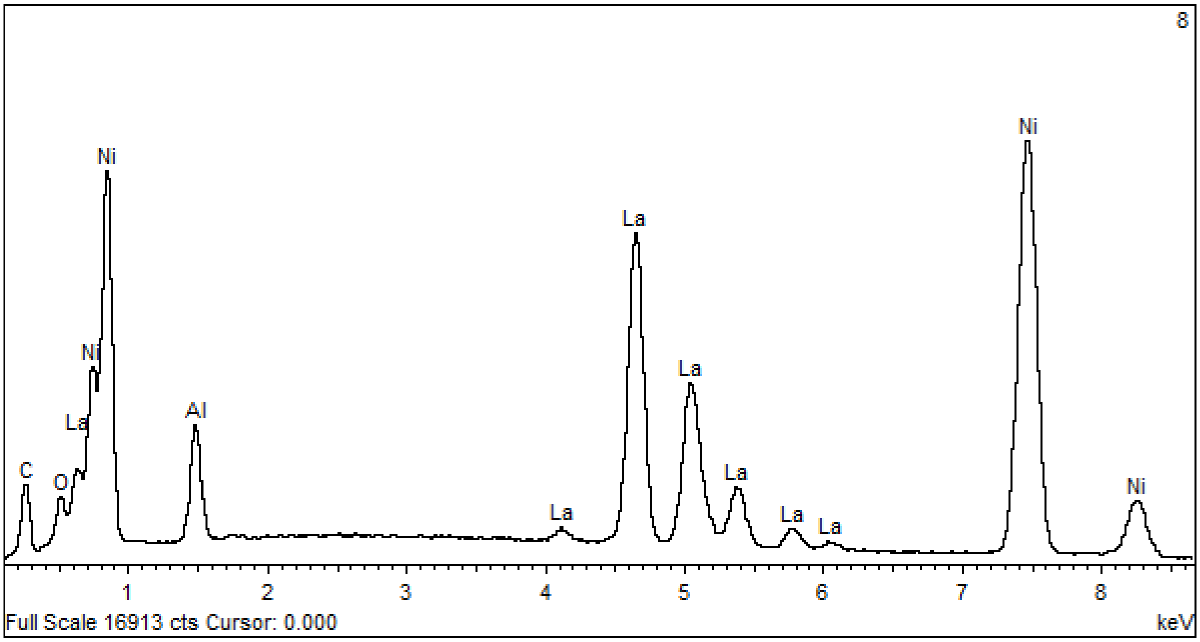
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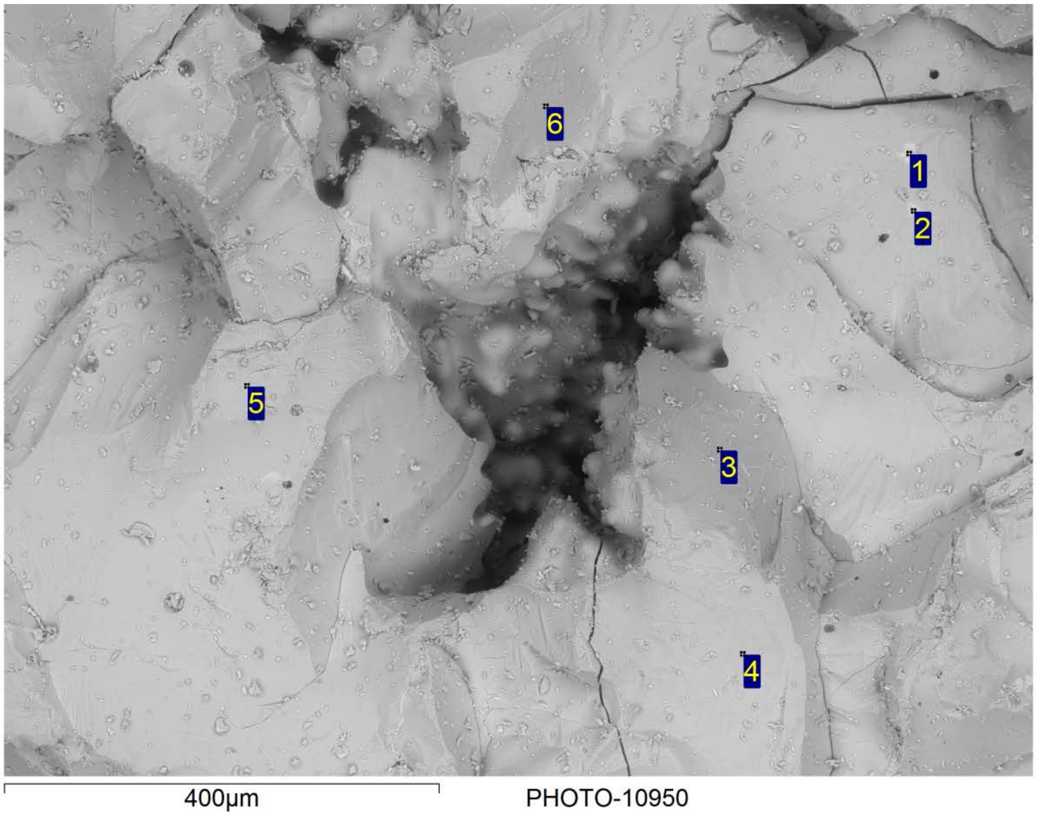
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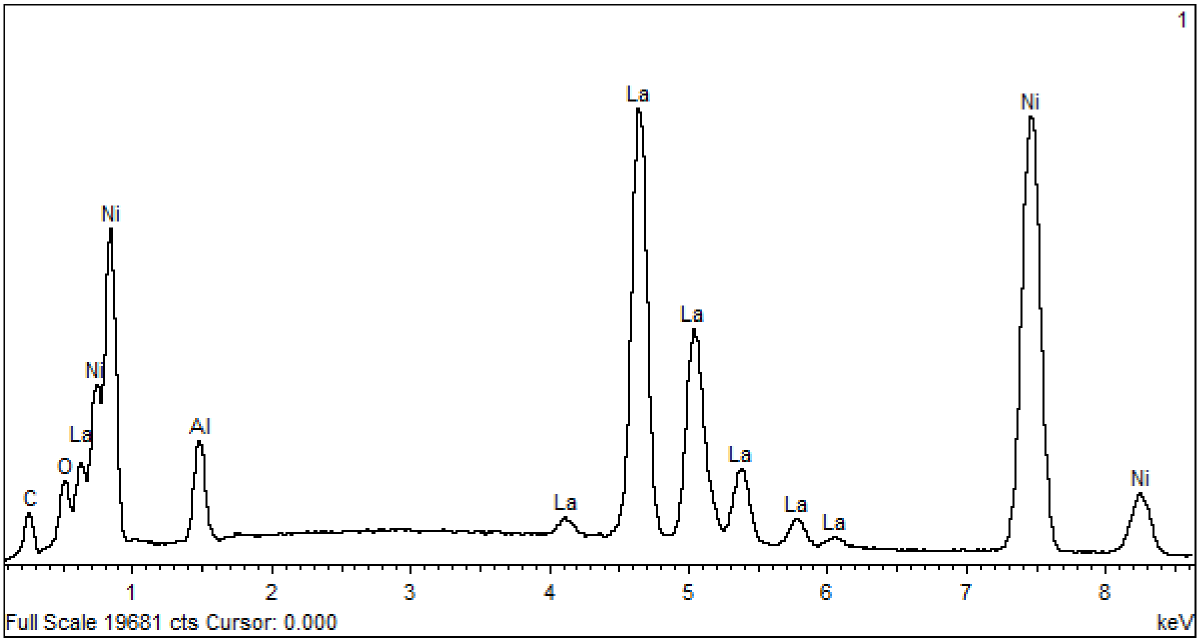
4
400µm
PHOTO-10950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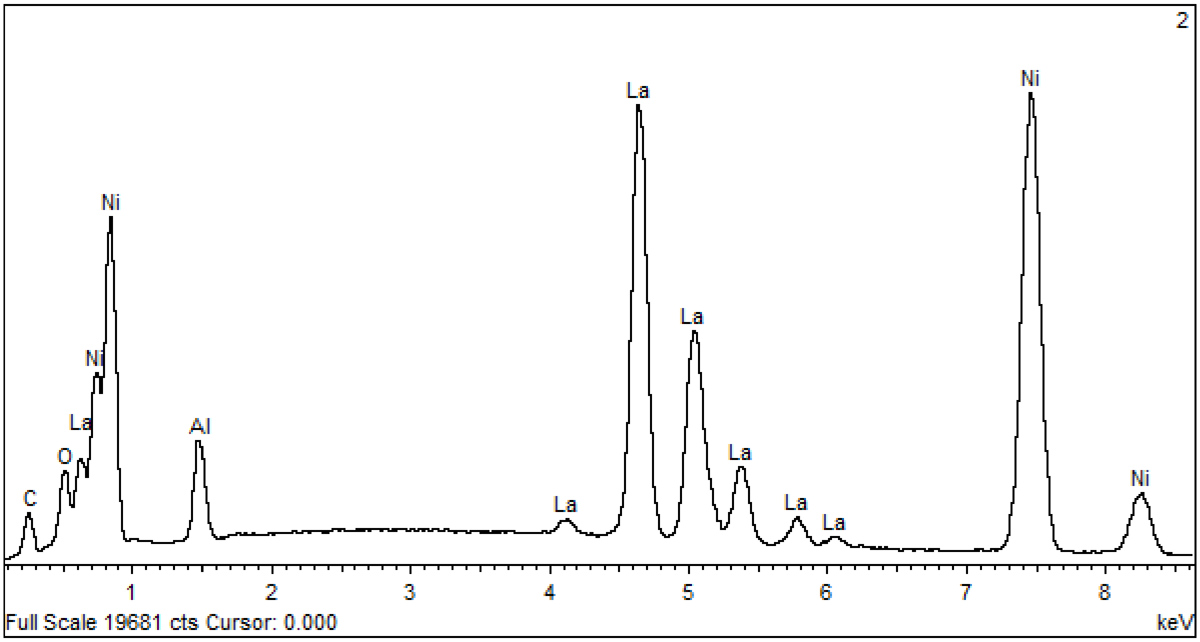
keV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La
Ni
La
O
Al
C
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Ni
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Full Scale 19681 cts Cursor: 0.000
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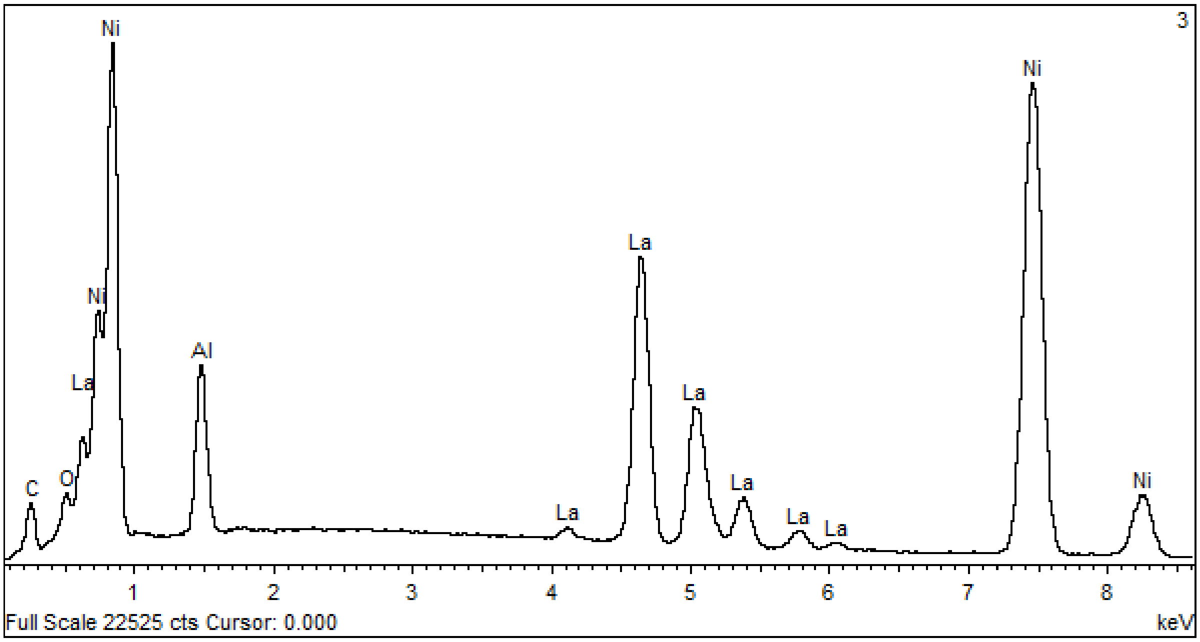
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La
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C
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La
La
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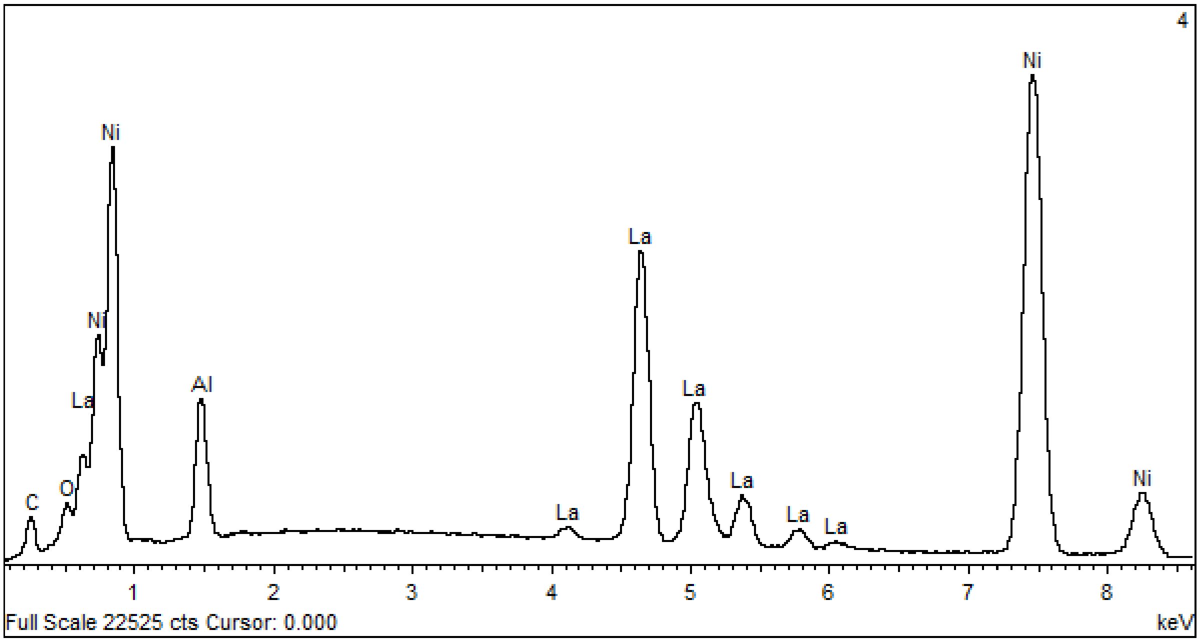
Full Scale 22525 cts Cursor: 0.000
keV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Ni
La
Al
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O
La
La
La
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Ni
Ni
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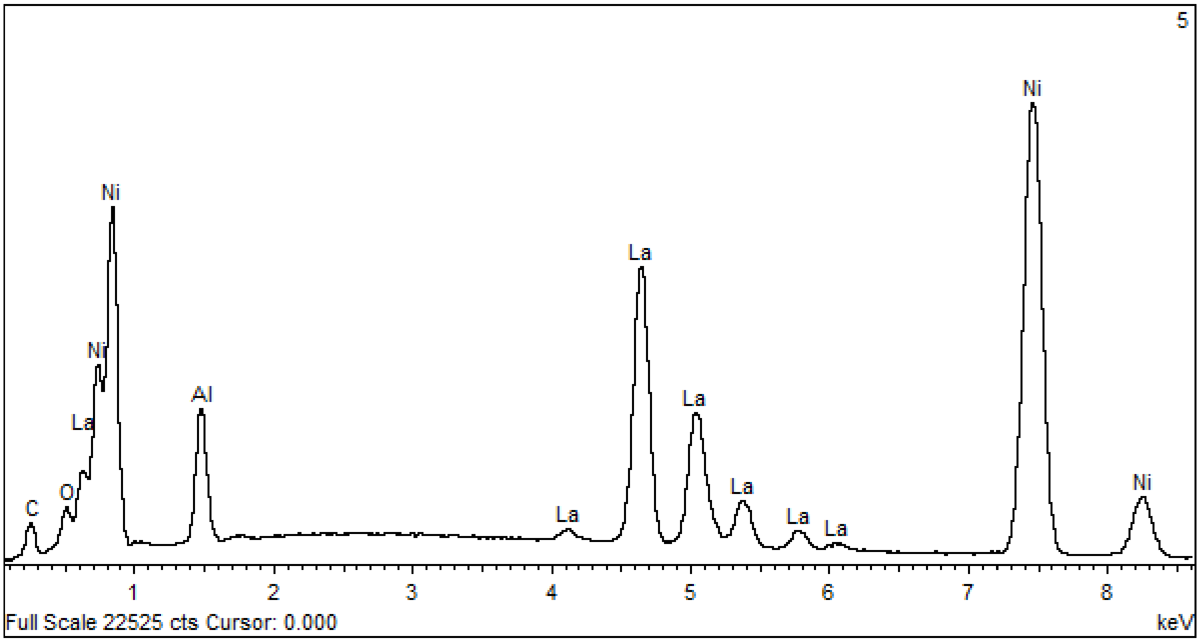
Full Scale 22525 cts Cursor: 0.000
keV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Ni
La
Al
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O
La
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La
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Ni
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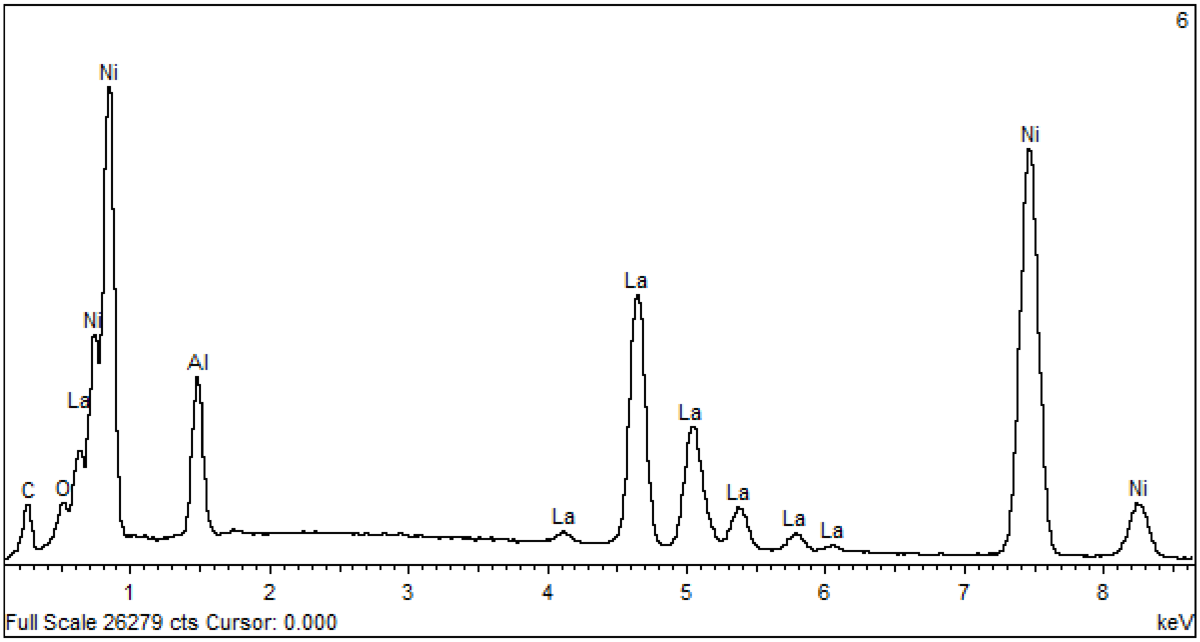
Full Scale 22525 cts Cursor: 0.000
keV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C
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La
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La
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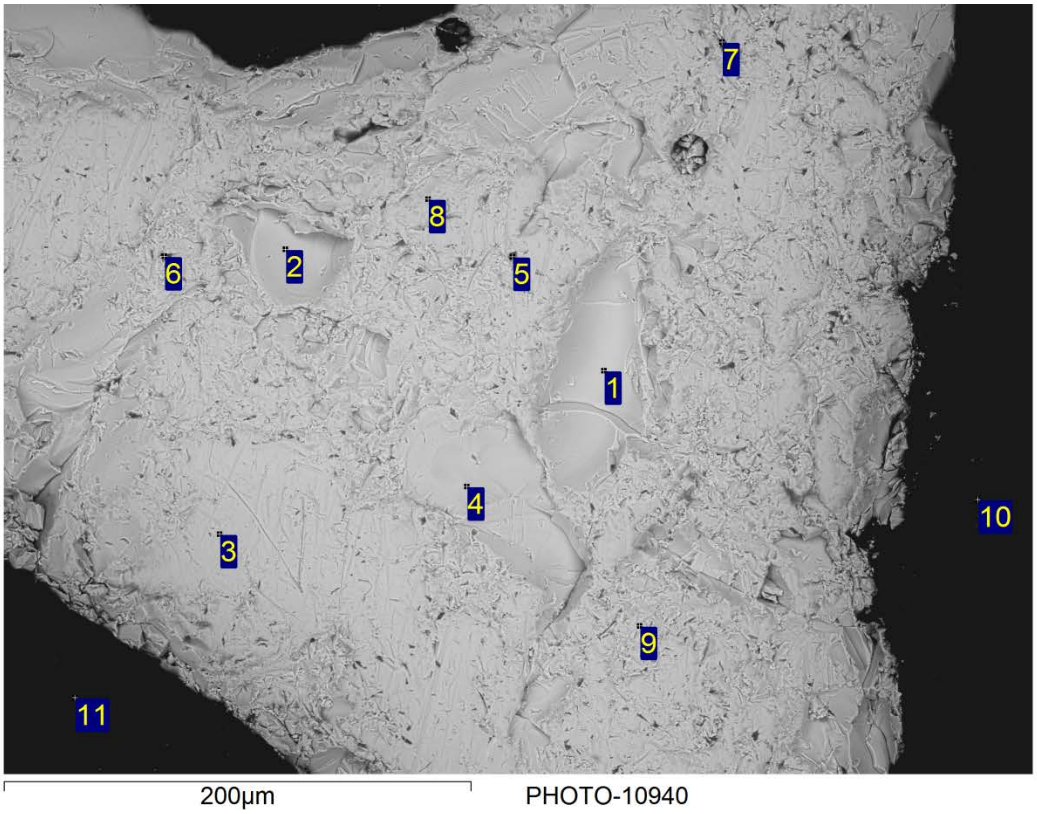
5
6
7
8
Full Scale 26279 cts Cursor: 0.000
keV

LANA 1684 Cross section

Energy dispersive spectroscopy (EDS) and scanning electron microscope (SEM) imaging were performed on a LANA sample mounted in epoxy and cross sectioned, without carbon coating. SEM imaging and EDS conditions were 20 kV, variable pressure = 66 Pa nitrogen. Because the EDS was not performed at high vacuum, there is some overlap in spectra between regions due to the scattering of the electron beam by nitrogen. In the LANA sample we see some silicon due to silicon carbide grit embedded in LANA (this can be seen as the many dark spots embedded in the LANA), which are shown as closeups in the EDS. There is also carbon and oxygen in the LANA Spots, due to the diffuse nature of the x-ray generating region, which may allow carbon from the epoxy to be detected, as well as the "adventitious" carbon film seen on most items not processed and analyzed in ultrahigh vacuum. The oxygen seen in all spectra may be due to residual oxygen in the SEM chamber or may be part of that "adventitious" carbon film.

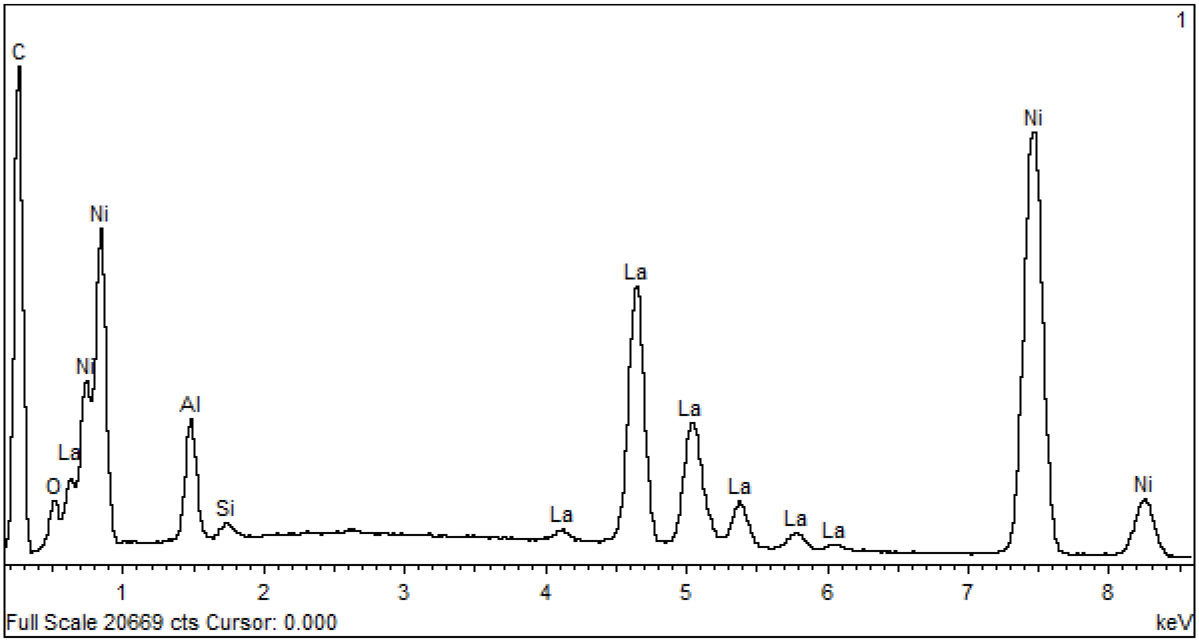


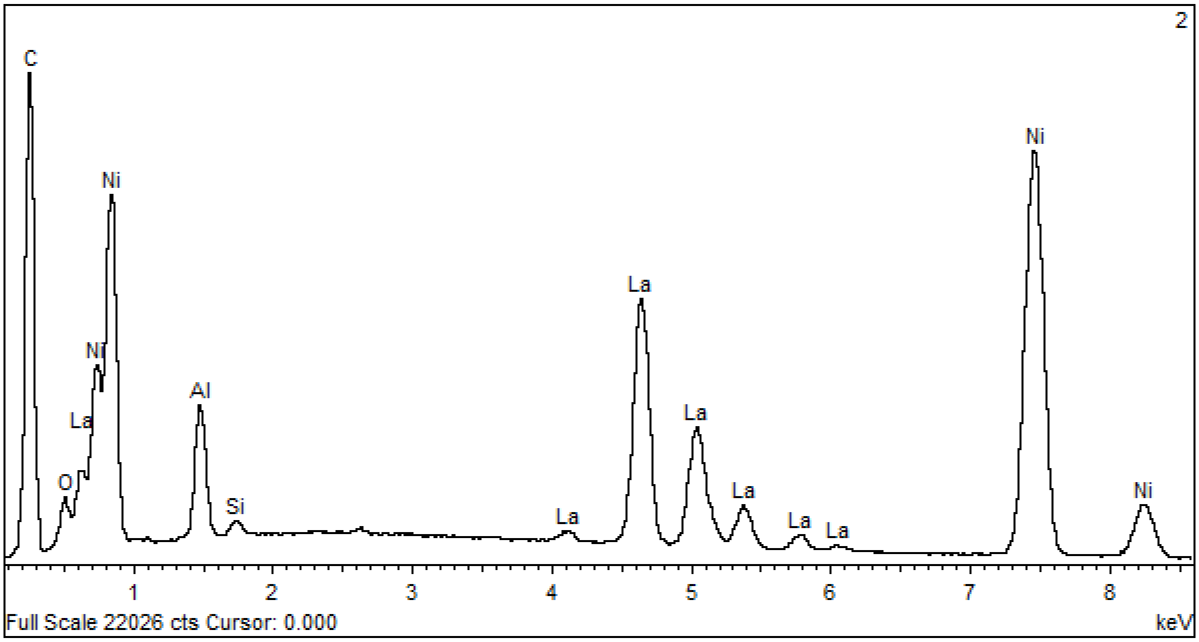
Spectra group themselves into:

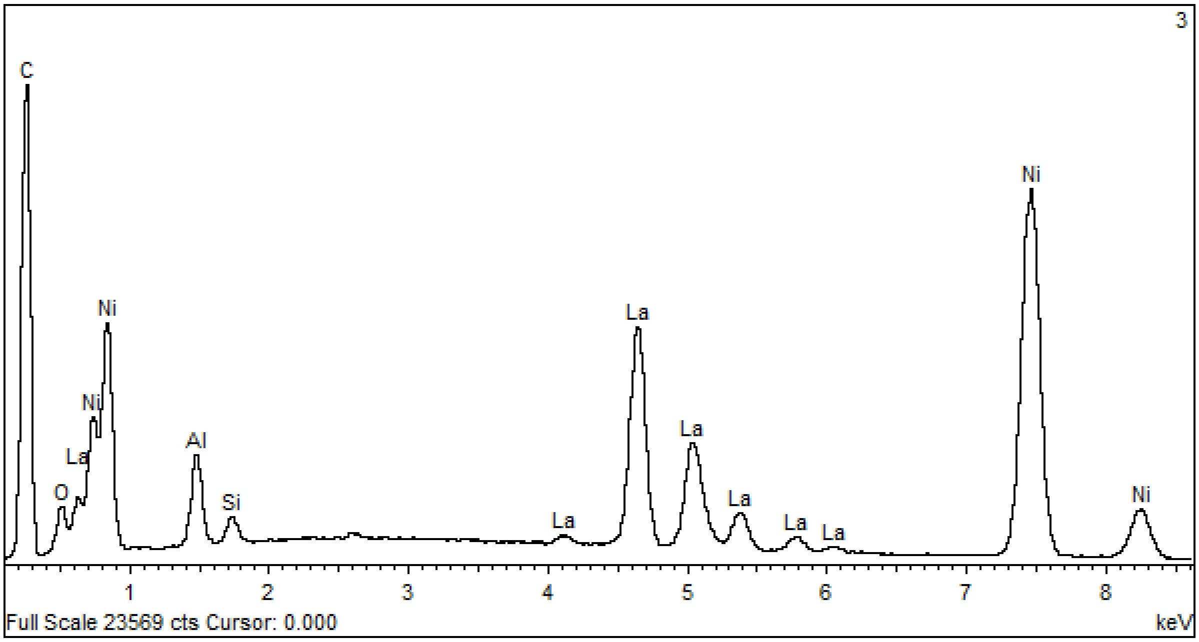
LANA Spots 1-4 and 8-9

SiC from polishing Spots 5-7 strongest element is silicon

Epoxy Spots 10-11 highest peak is carbon and includes traces of chlorine and sulfur



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C
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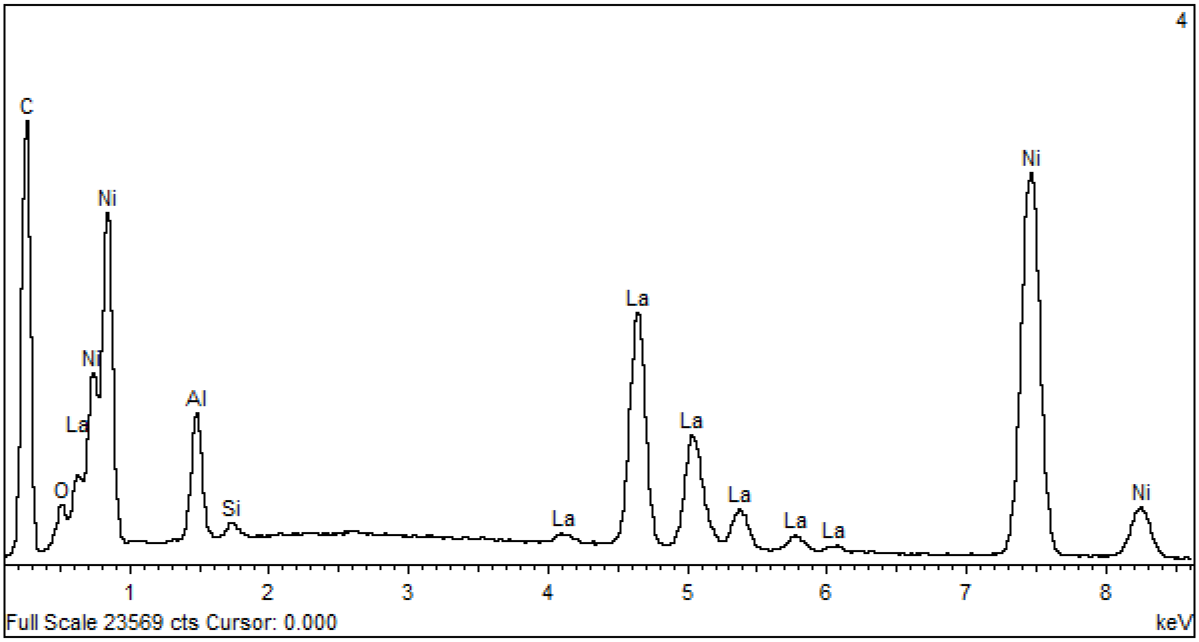
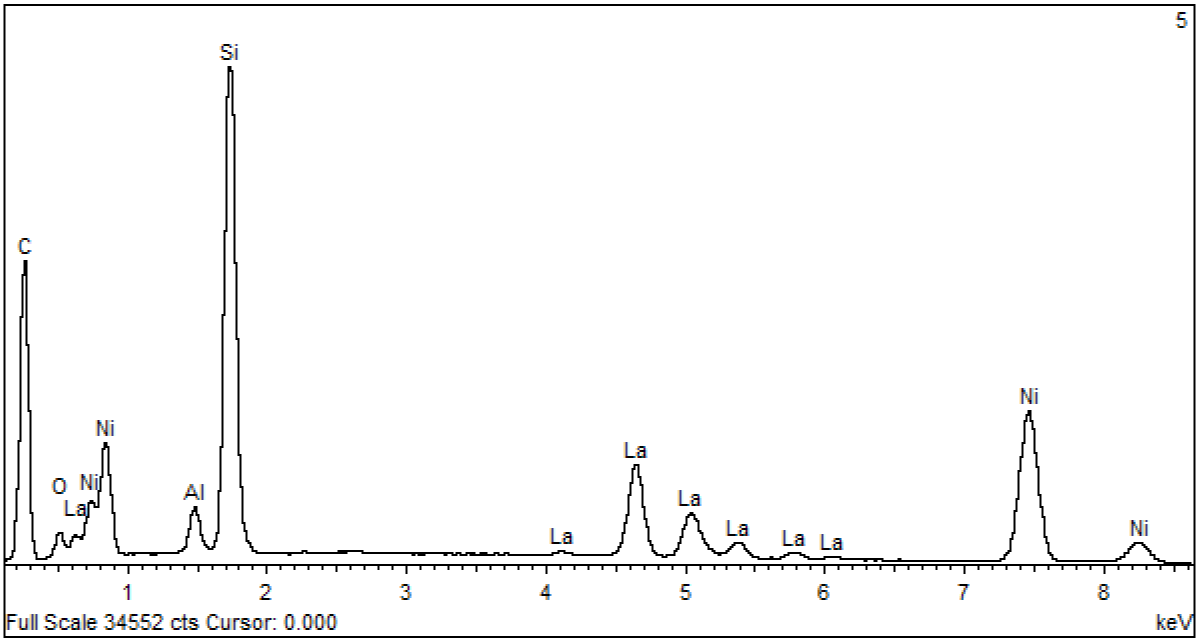
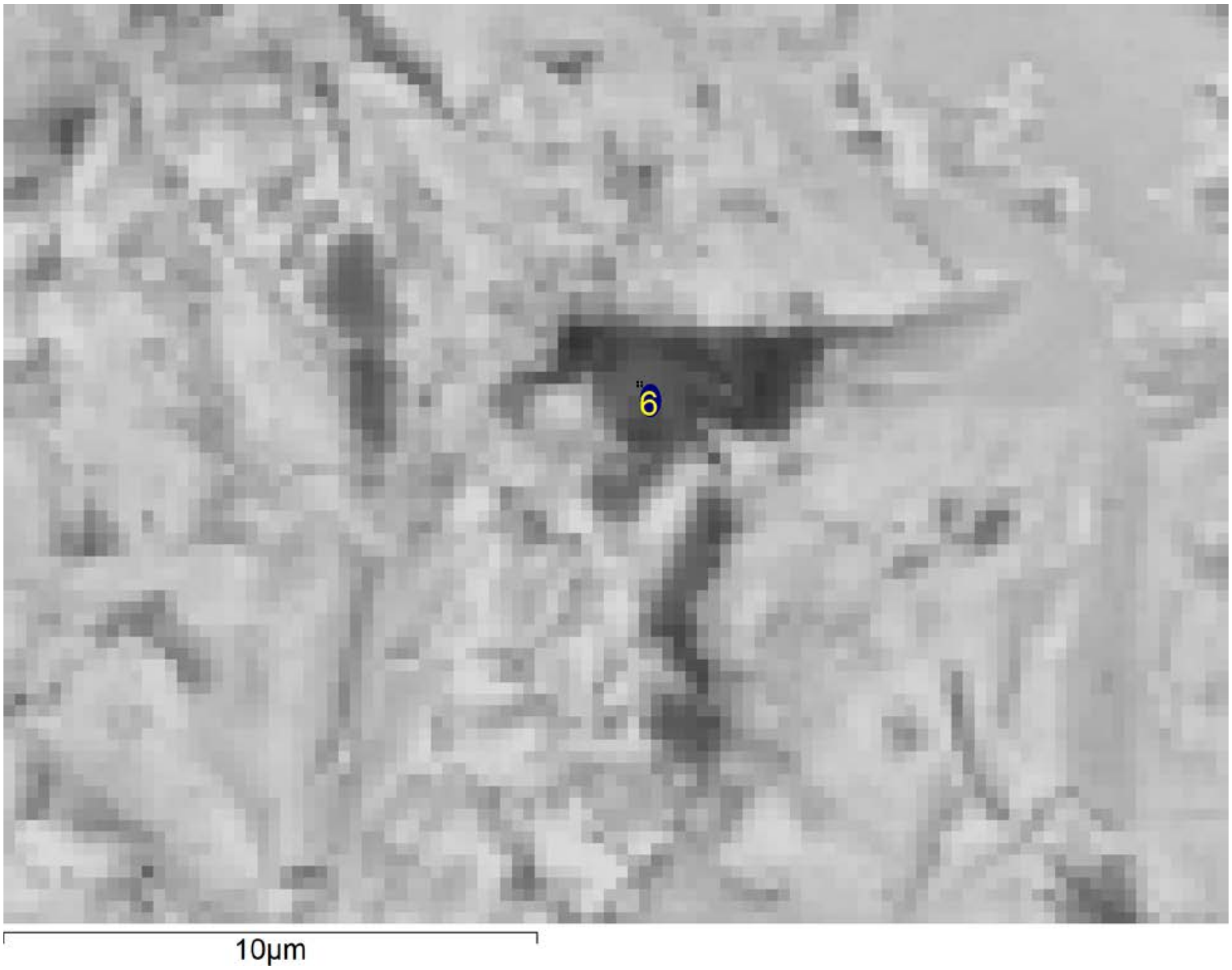
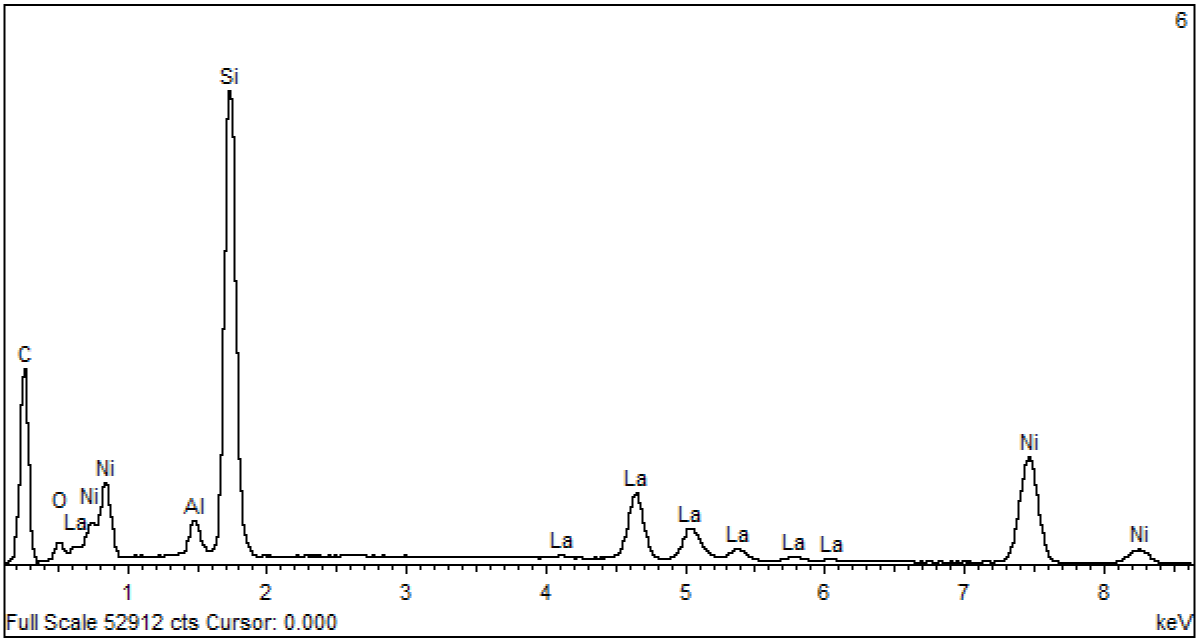
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 23569 cts Cursor: 0.000
keV

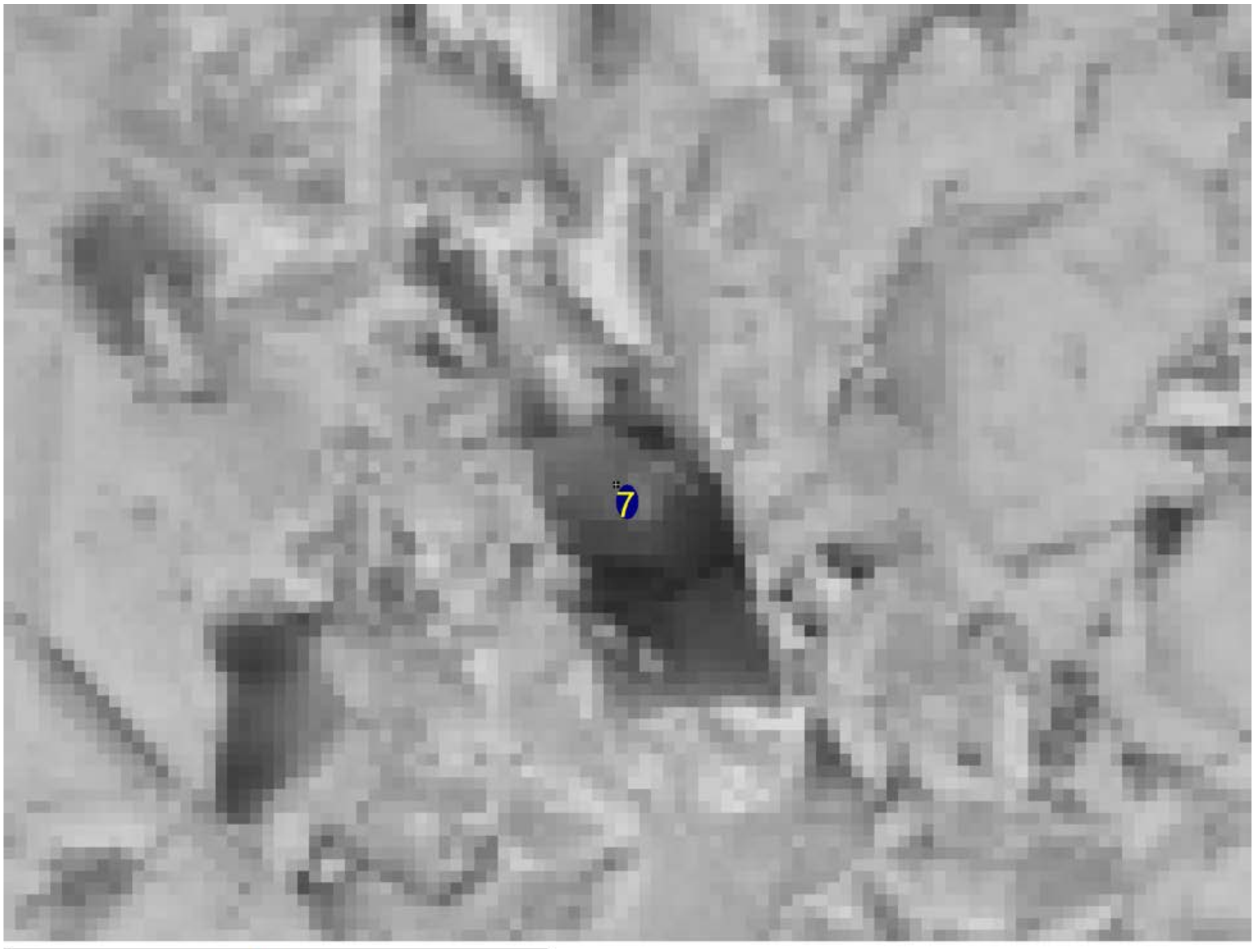

4
C
Ni
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 23569 cts Cursor: 0.000
keV

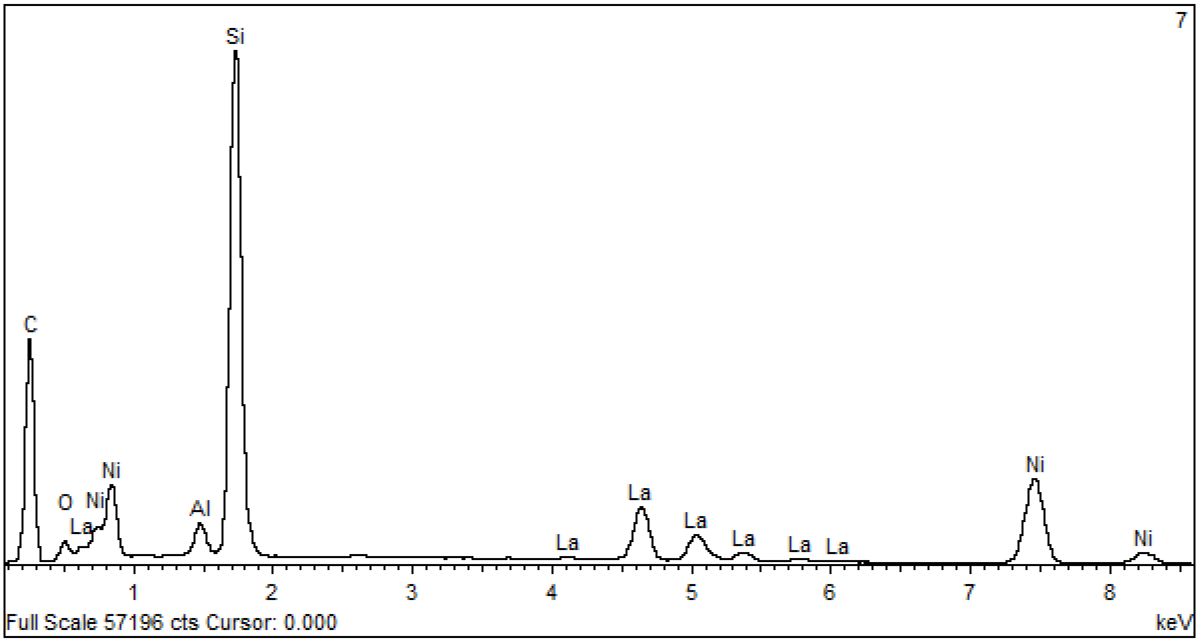

5
10µm

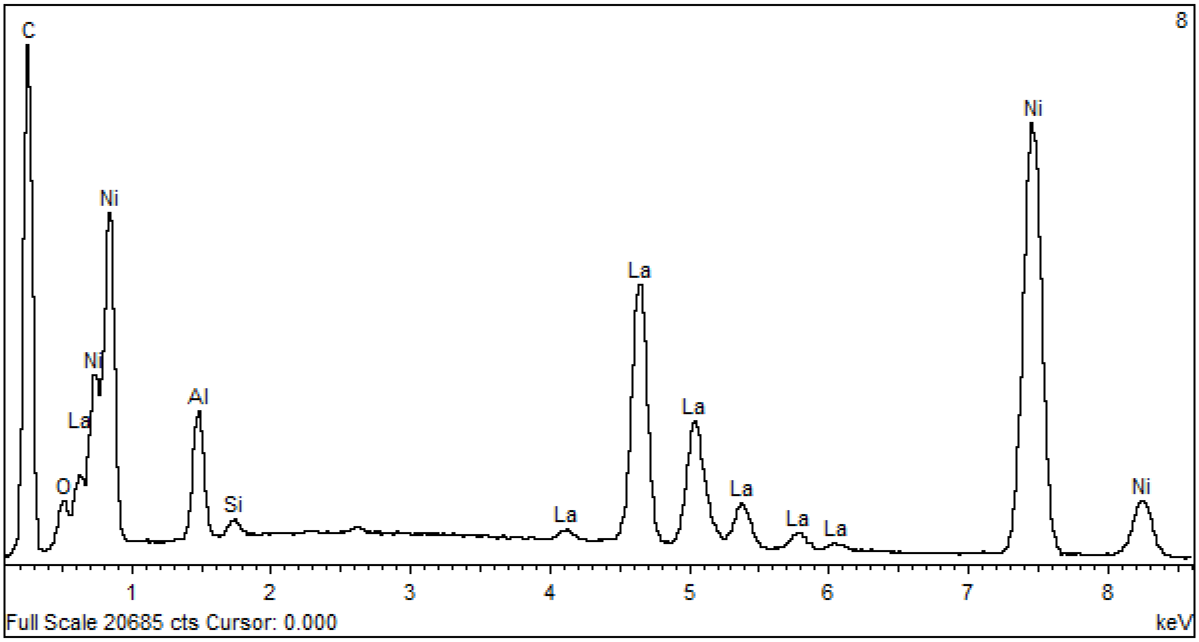

5
Si
C
Ni
O
Ni
La
Al
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 34552 cts Cursor: 0.000
keV

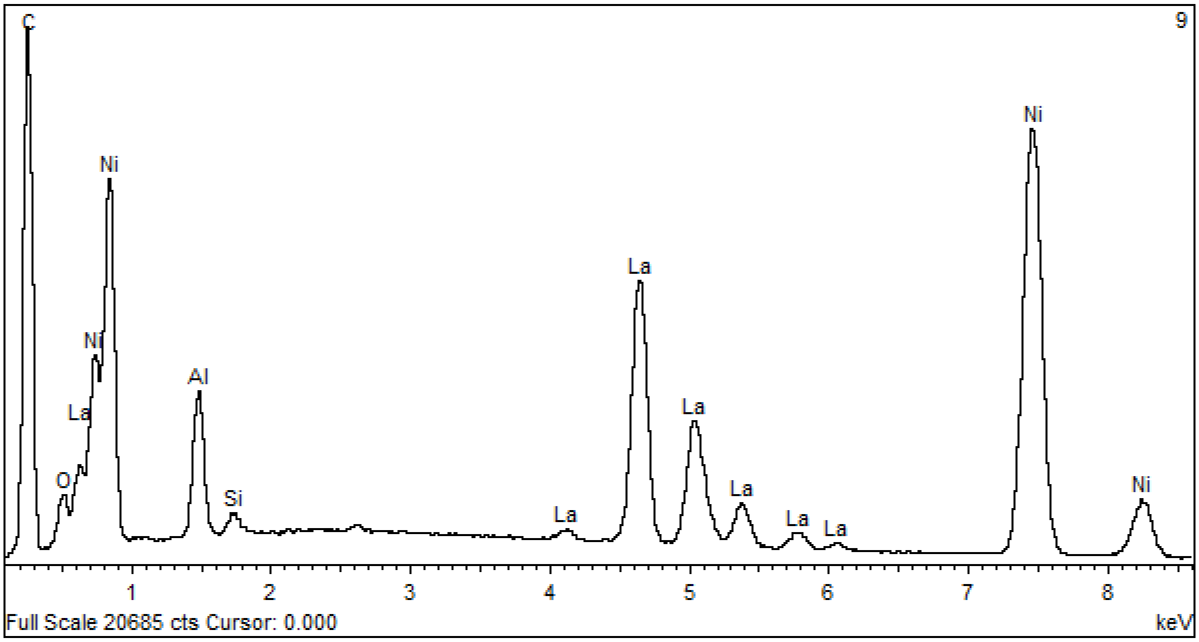

6
10μm

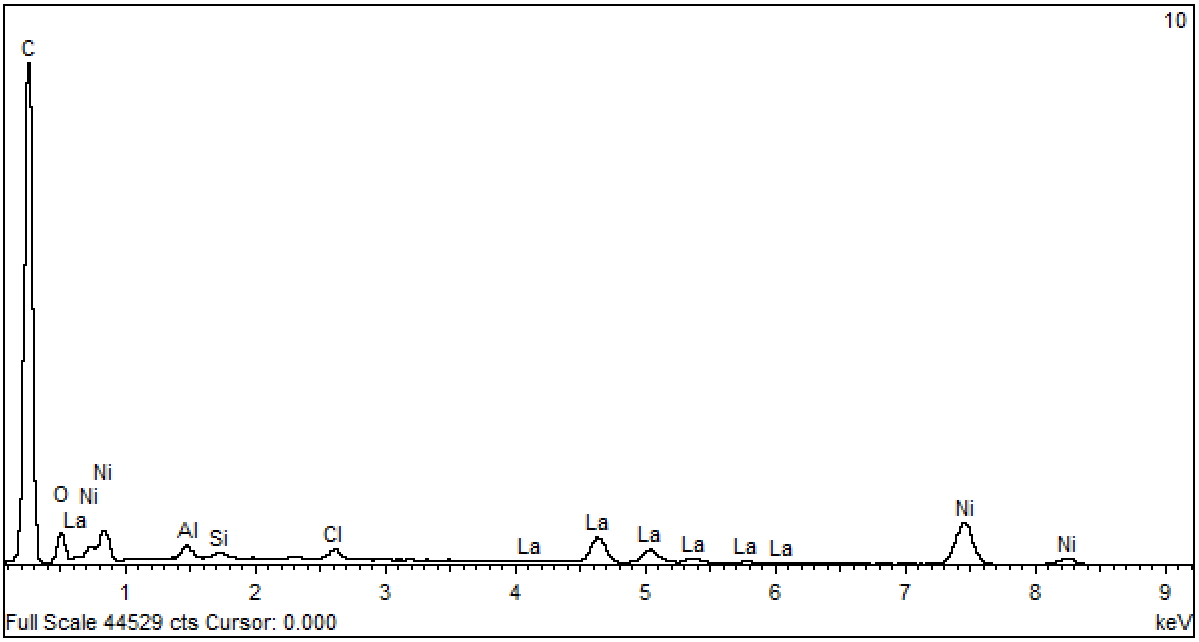

6
Si
C
O
Ni
La
Ni
Al
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 52912 cts Cursor: 0.000
keV

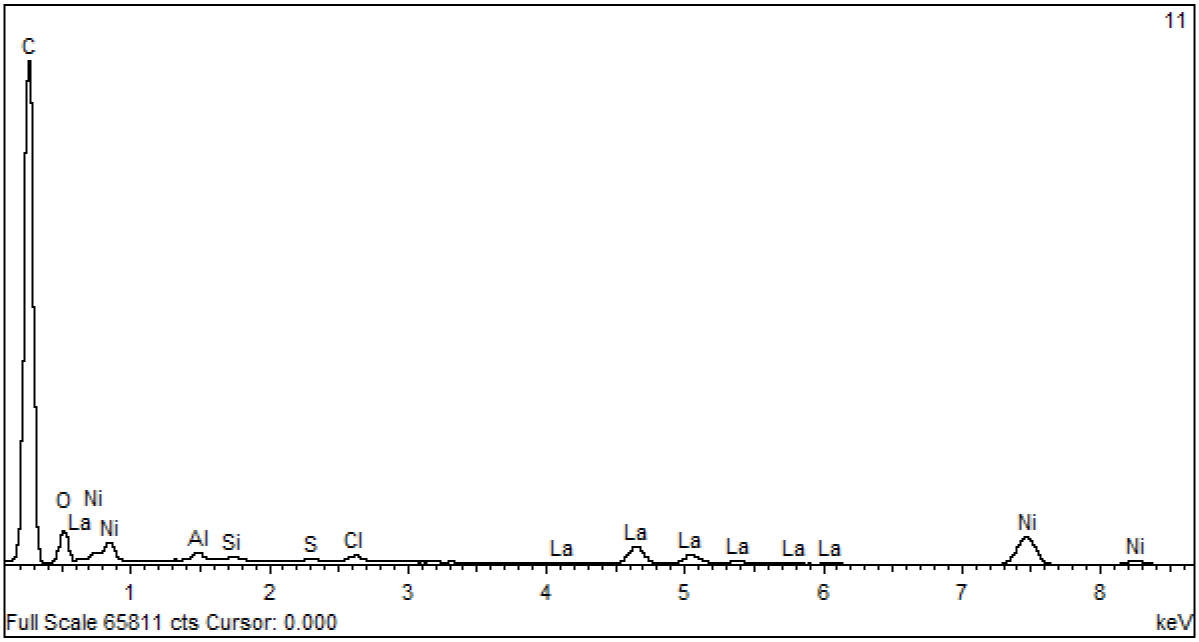

7
10µm

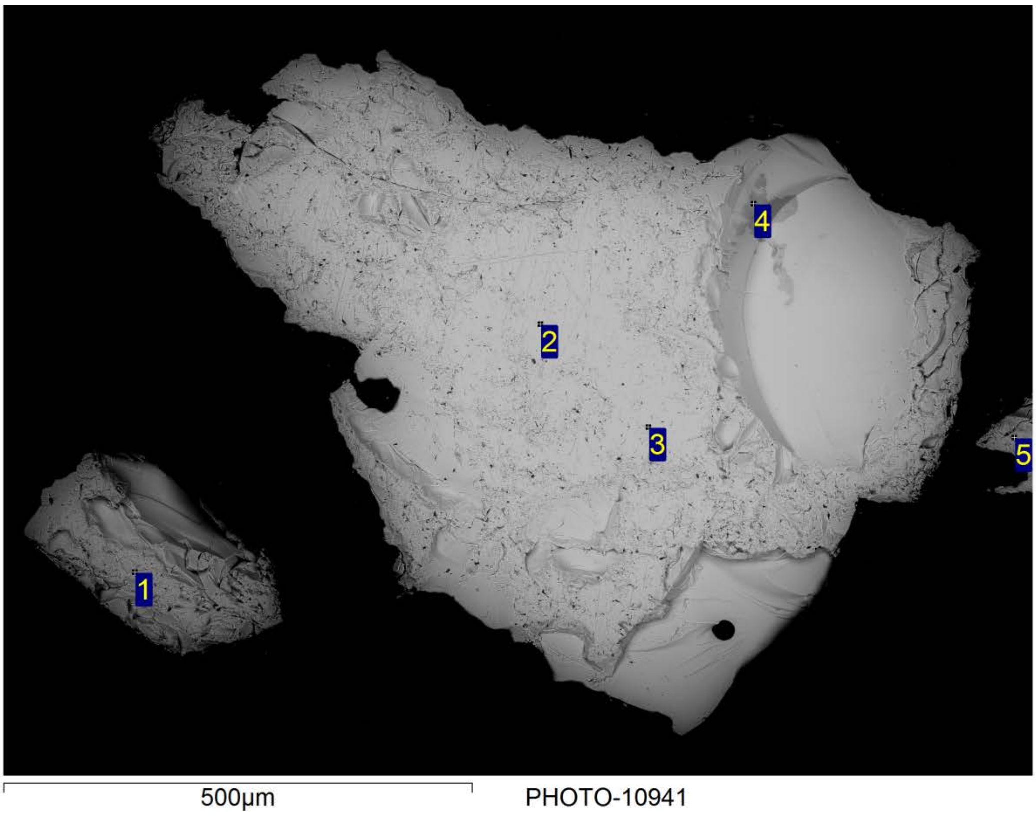

7
Si
C
Ni
O
Ni
La
Al
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 57196 cts Cursor: 0.000
keV


8
C
Ni
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 20685 cts Cursor: 0.000
keV

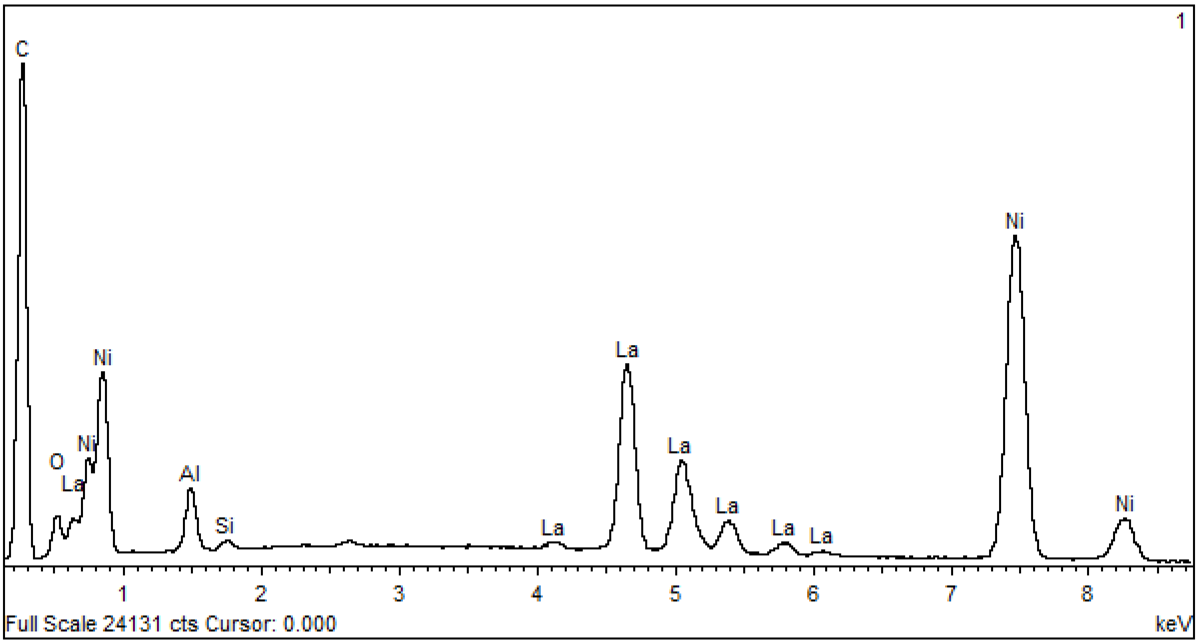

9
C
Ni
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 20685 cts Cursor: 0.000
keV

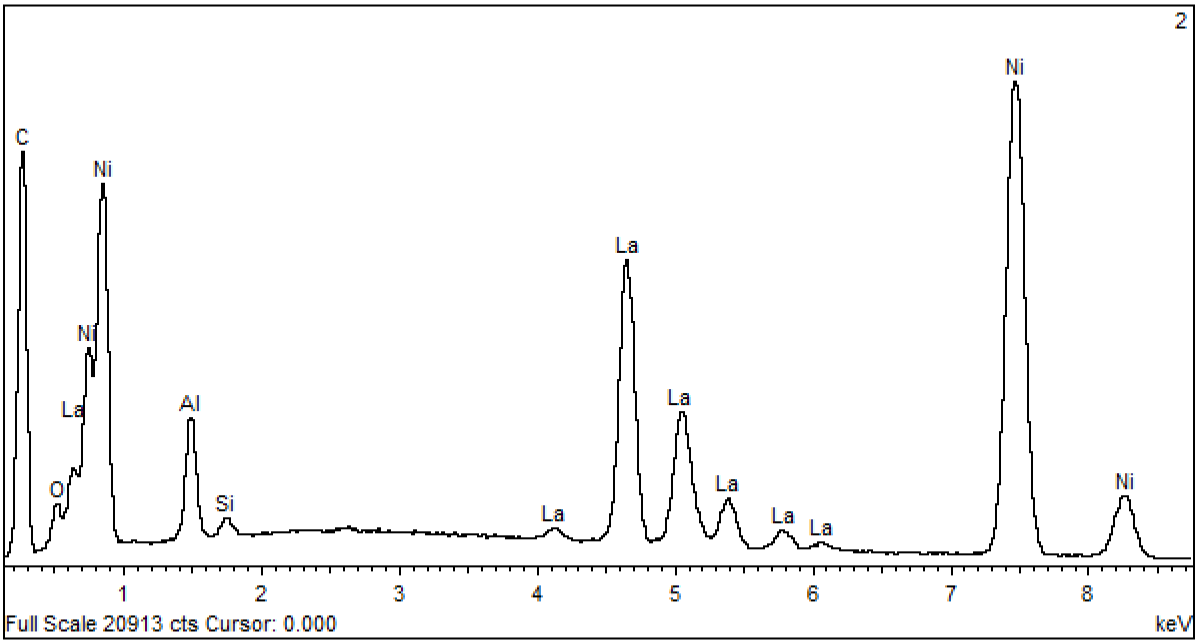

10
C
O
Ni
Ni
La
Al
Si
Cl
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
9
Full Scale 44529 cts Cursor: 0.000
keV

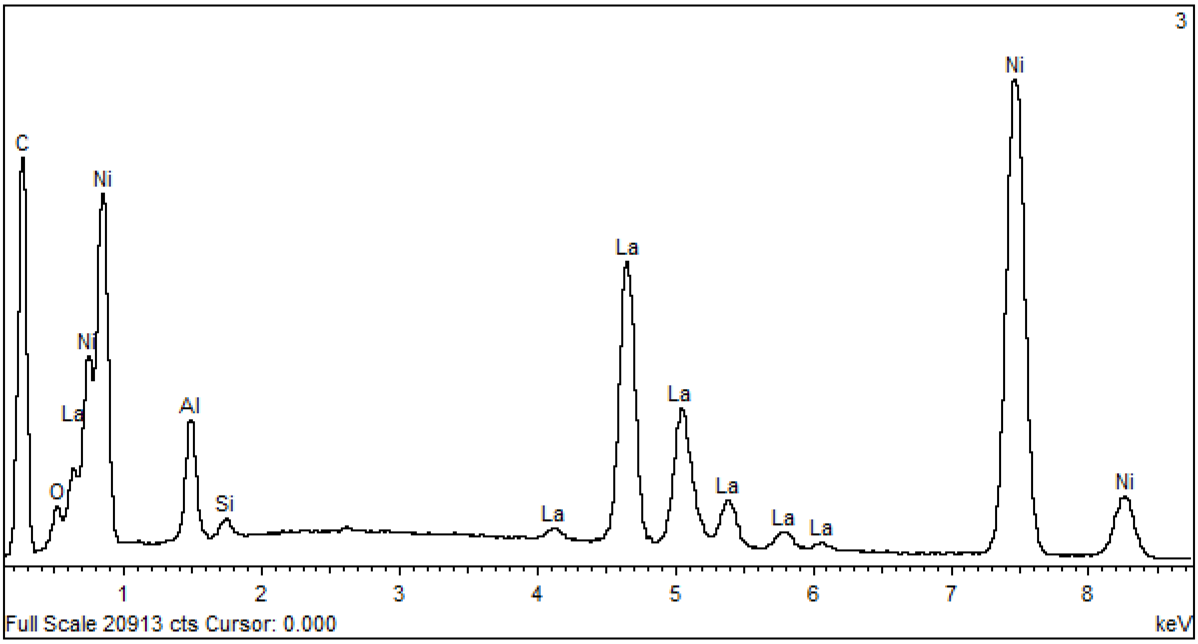

11
C
O
Ni
La
Ni
Al
Si
S
Cl
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 65811 cts Cursor: 0.000
keV

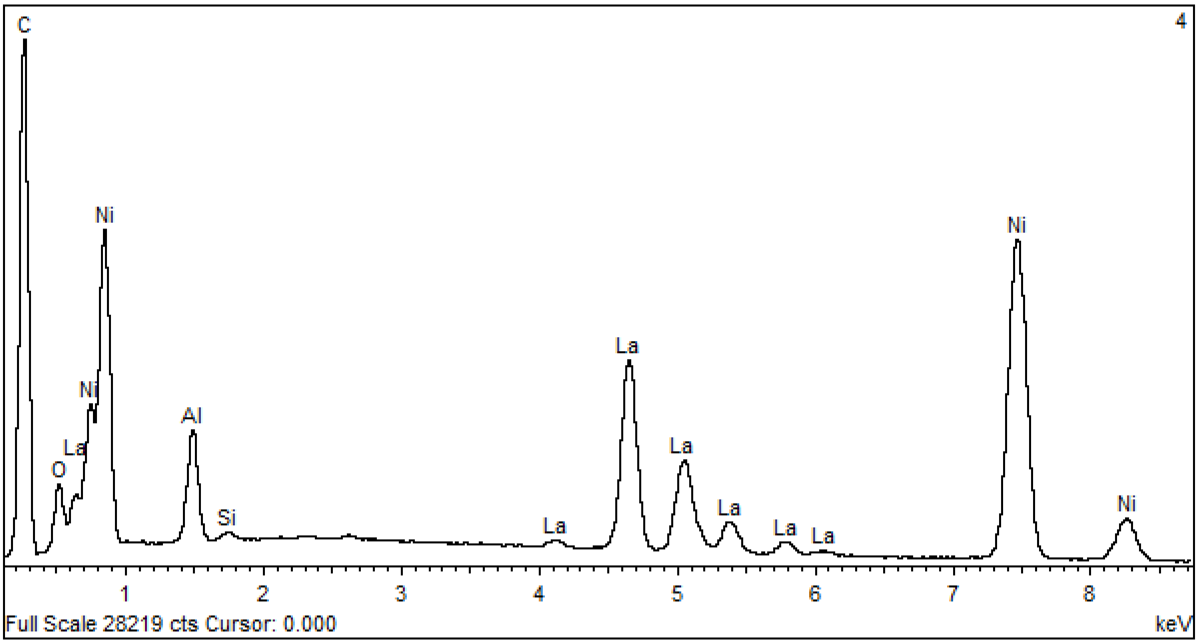


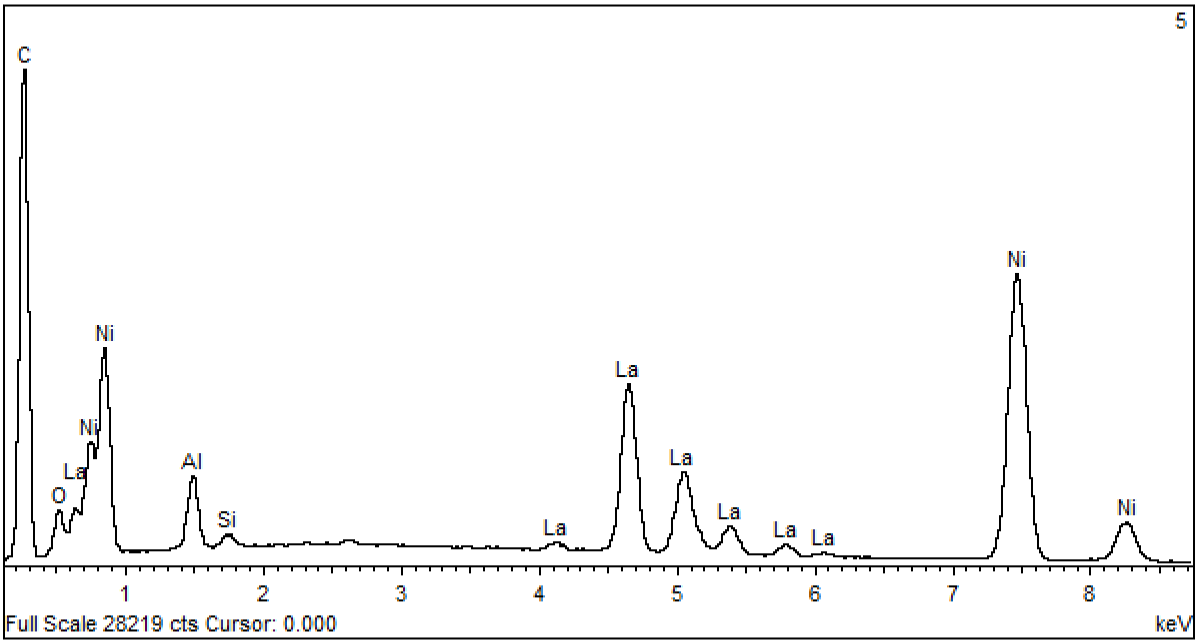
Was looking for different phase in Spot 4, looked the same as all the other spots, difference in color is probably due to heavier carbon contamination.



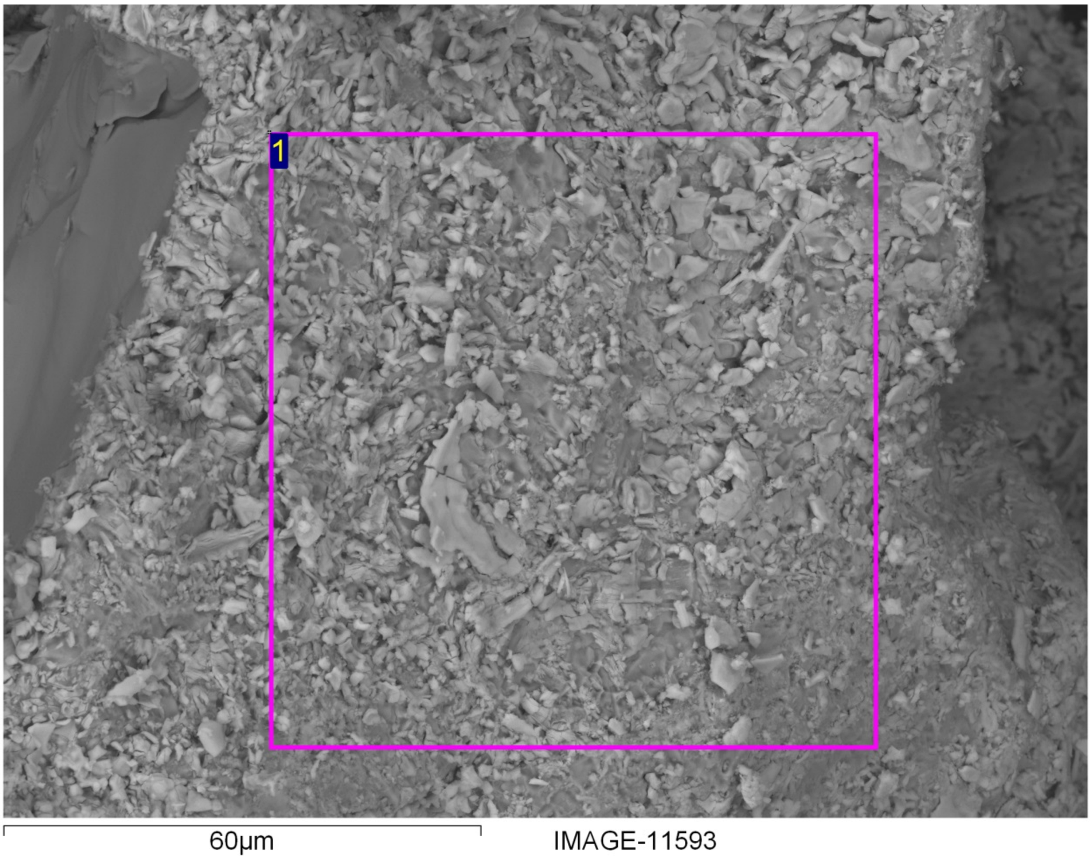

2
C
Ni
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 20913 cts Cursor: 0.000
keV


3
C
Ni
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 20913 cts Cursor: 0.000
keV

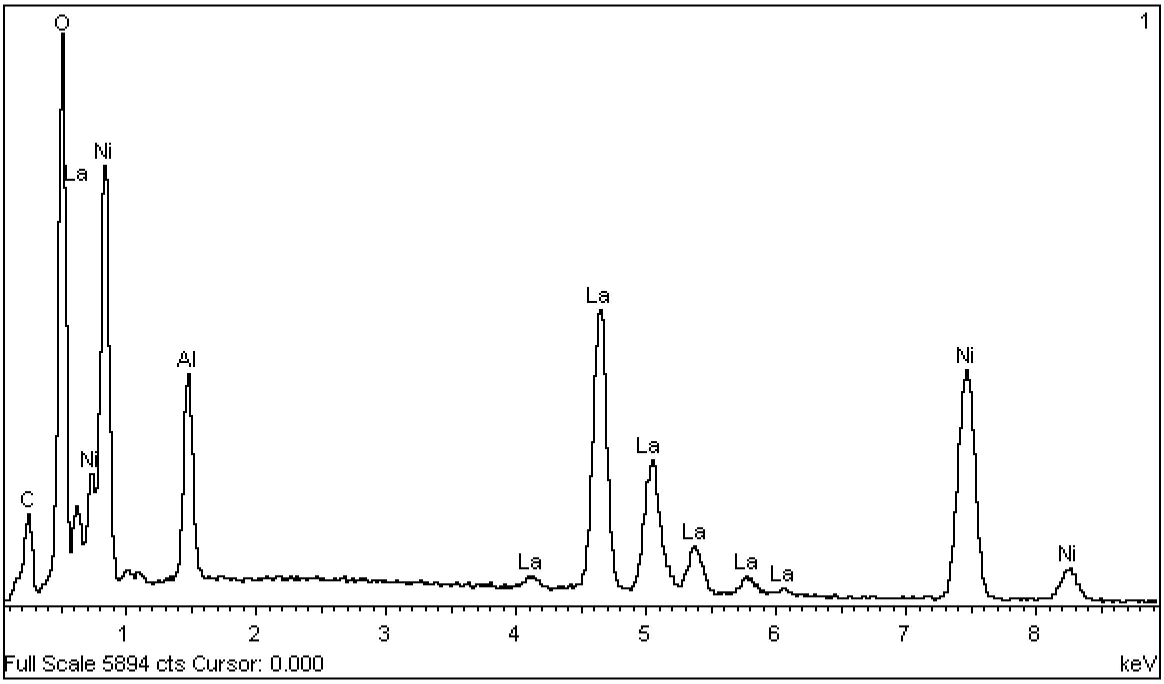

4
C
Ni
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 28219 cts Cursor: 0.000
keV

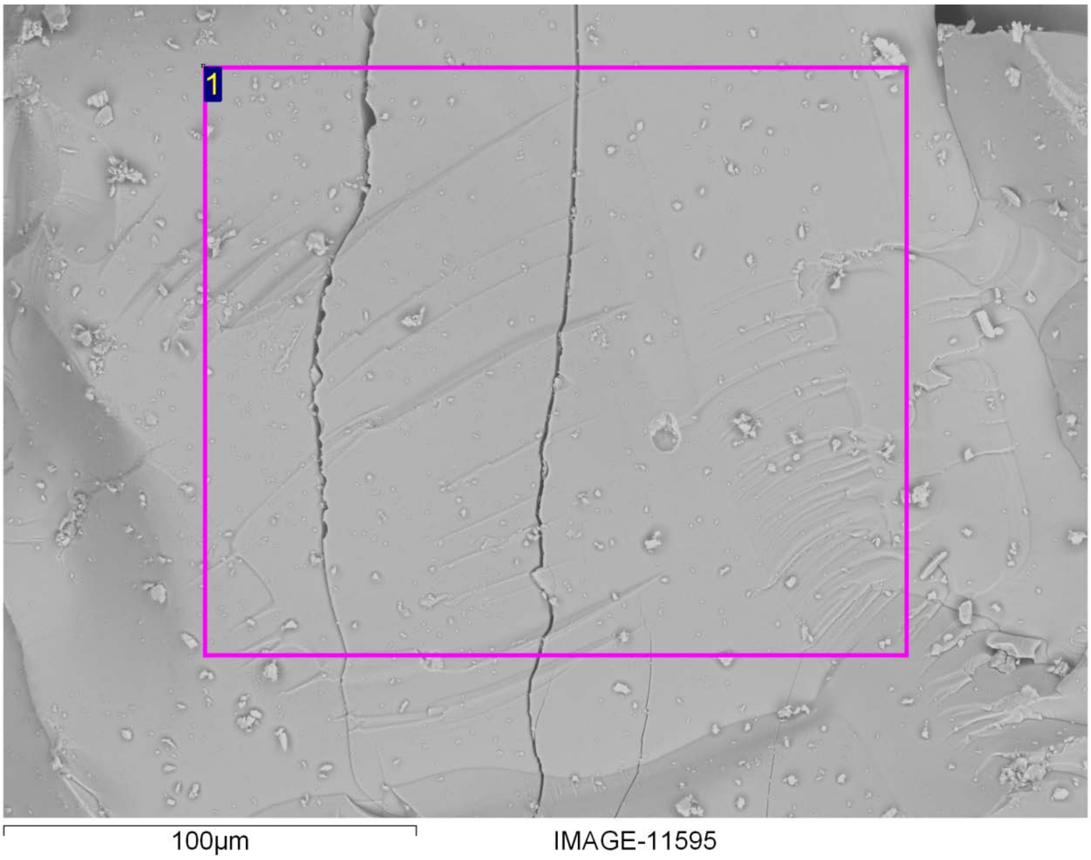

5
C
Ni
Ni
La
O
Al
Si
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 28219 cts Cursor: 0.000
keV

Part 2. LANA85 Material - Particles LaNi4.15Al0.85 3683, Report from H. Ajo, ADS

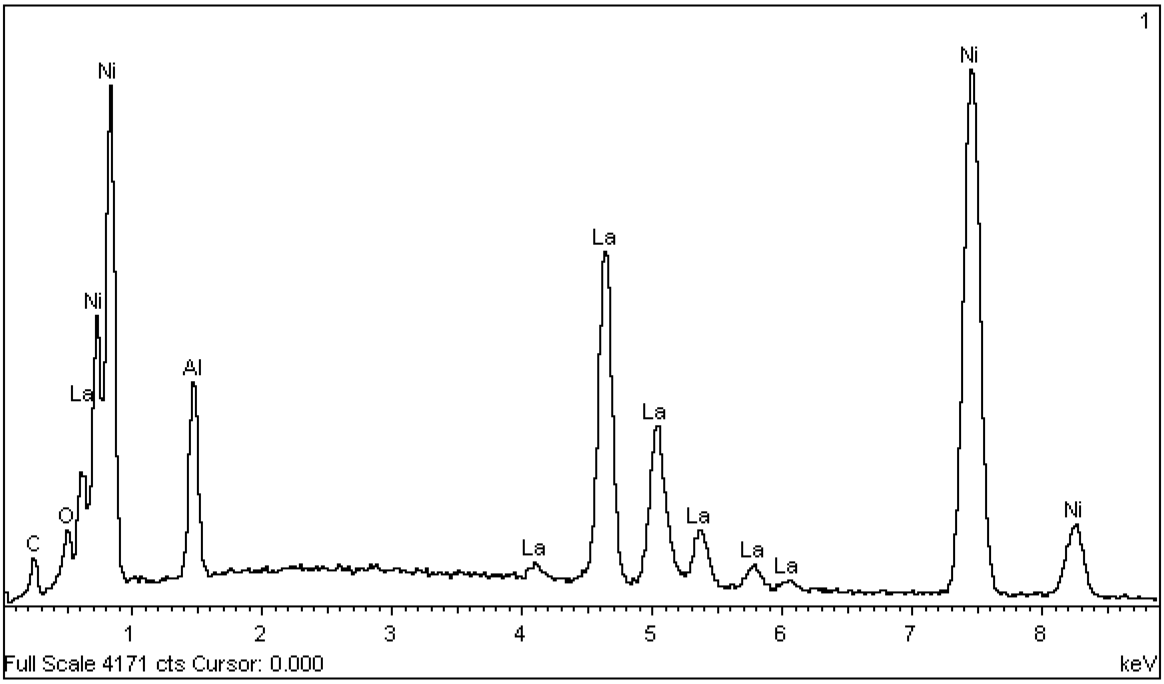


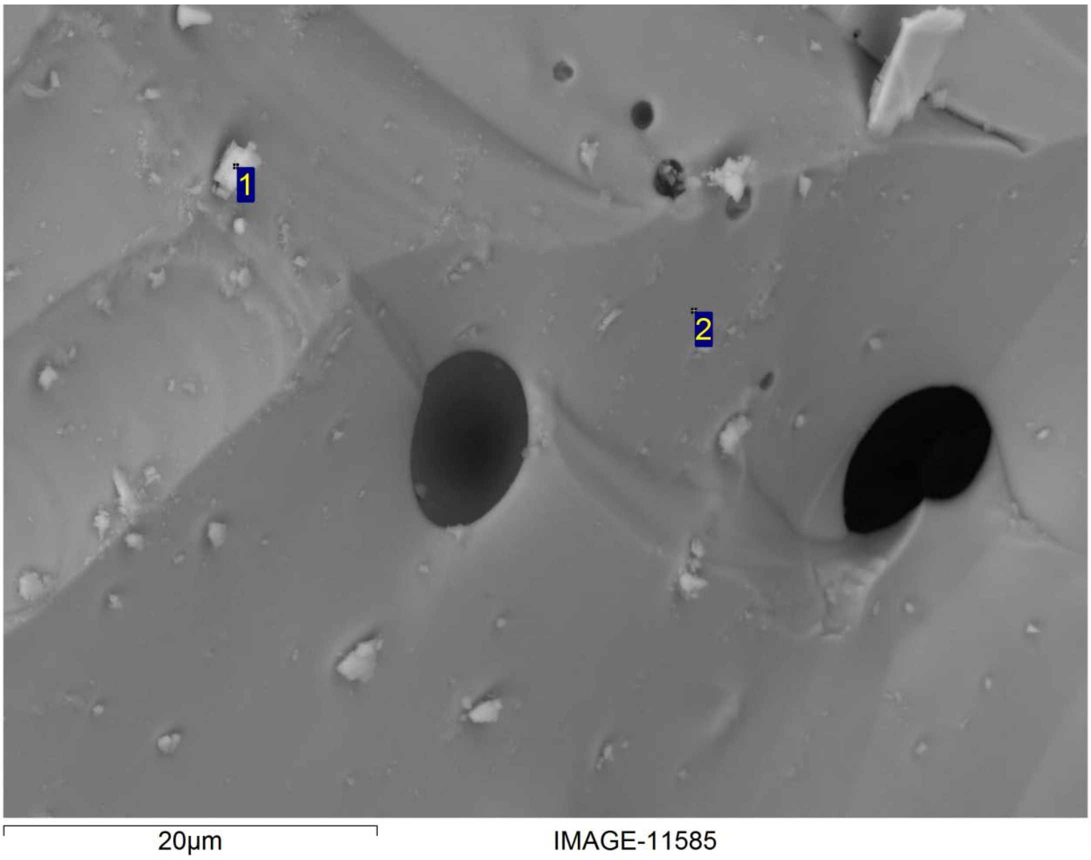
Note: `1` is an area.

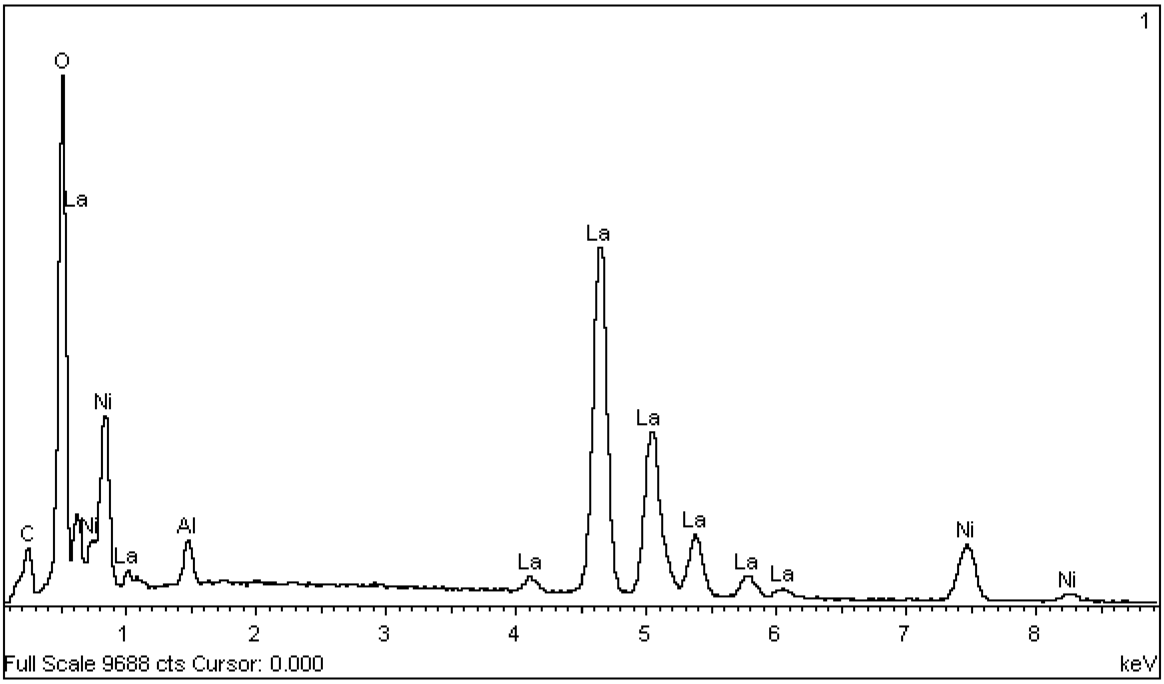


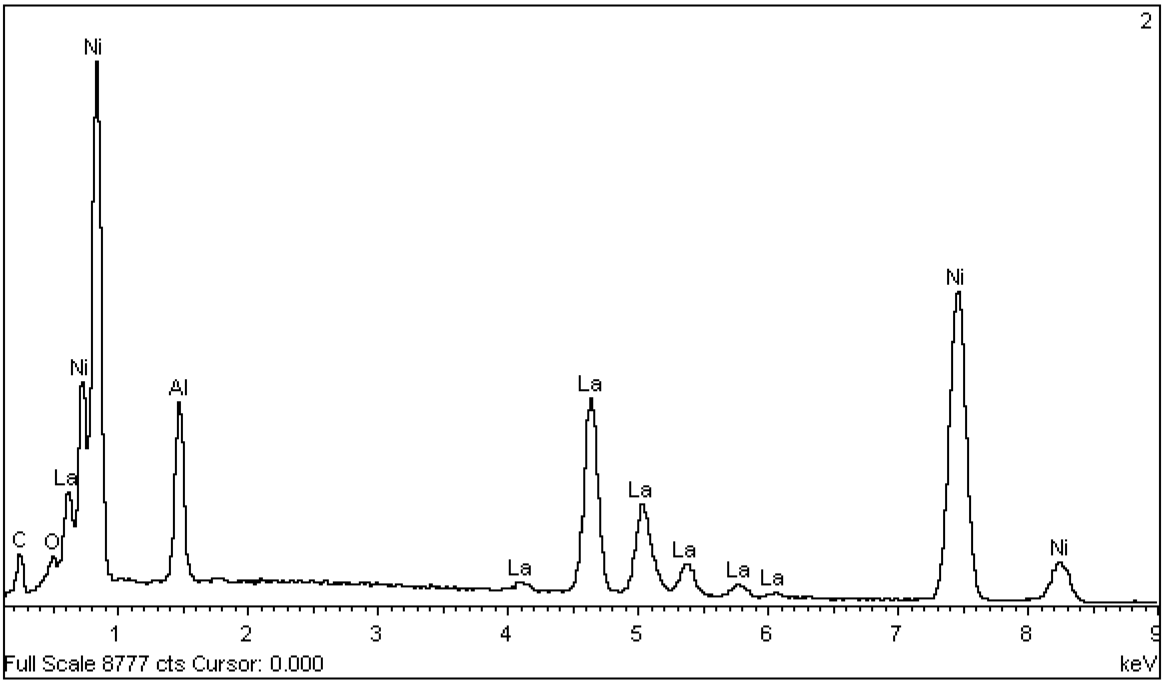


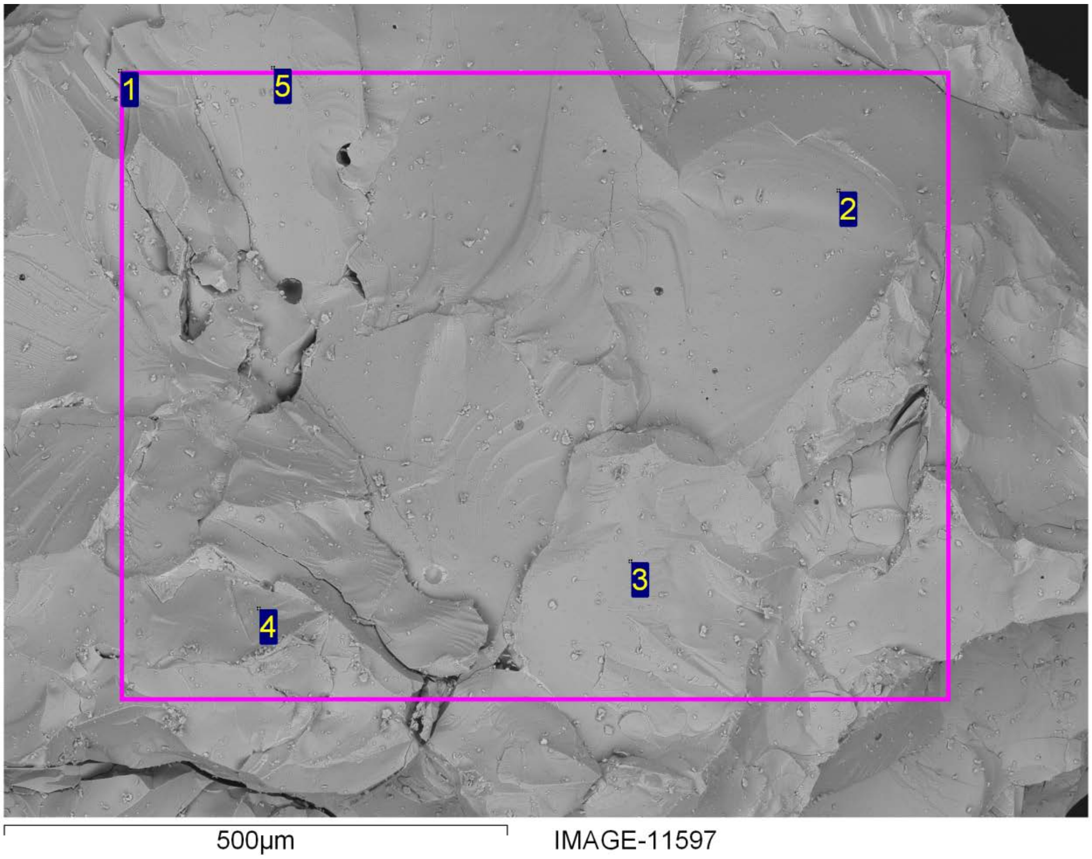
Note: `1` is an area.



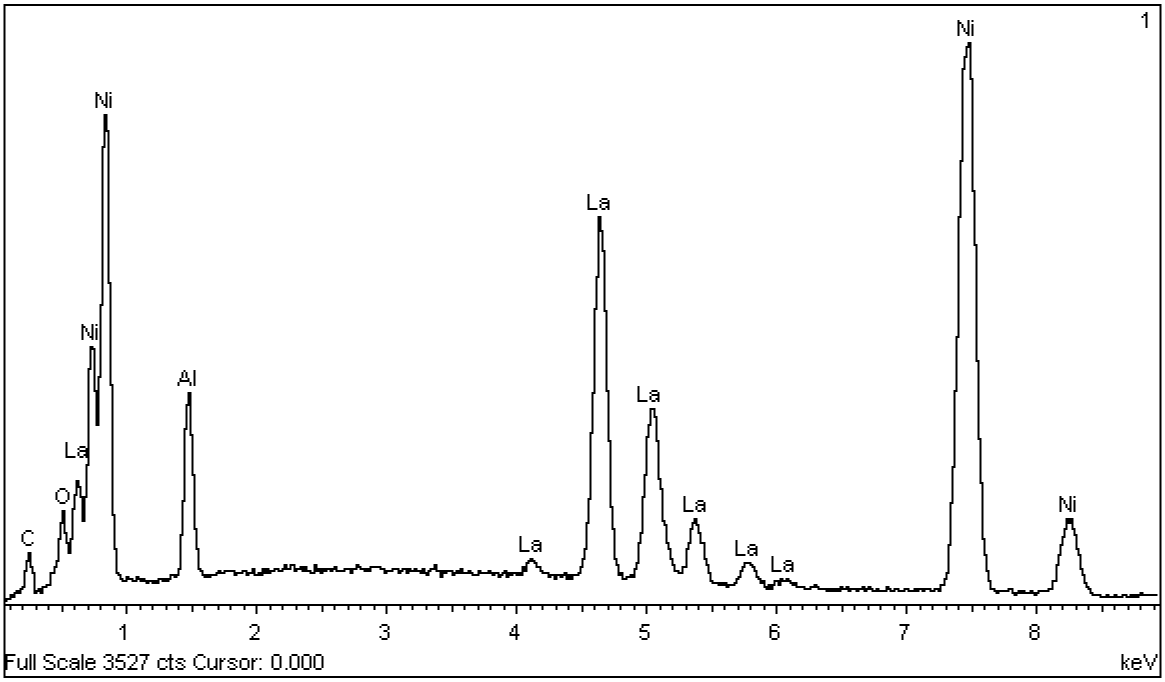

1
2
20µm
IMAGE-11585

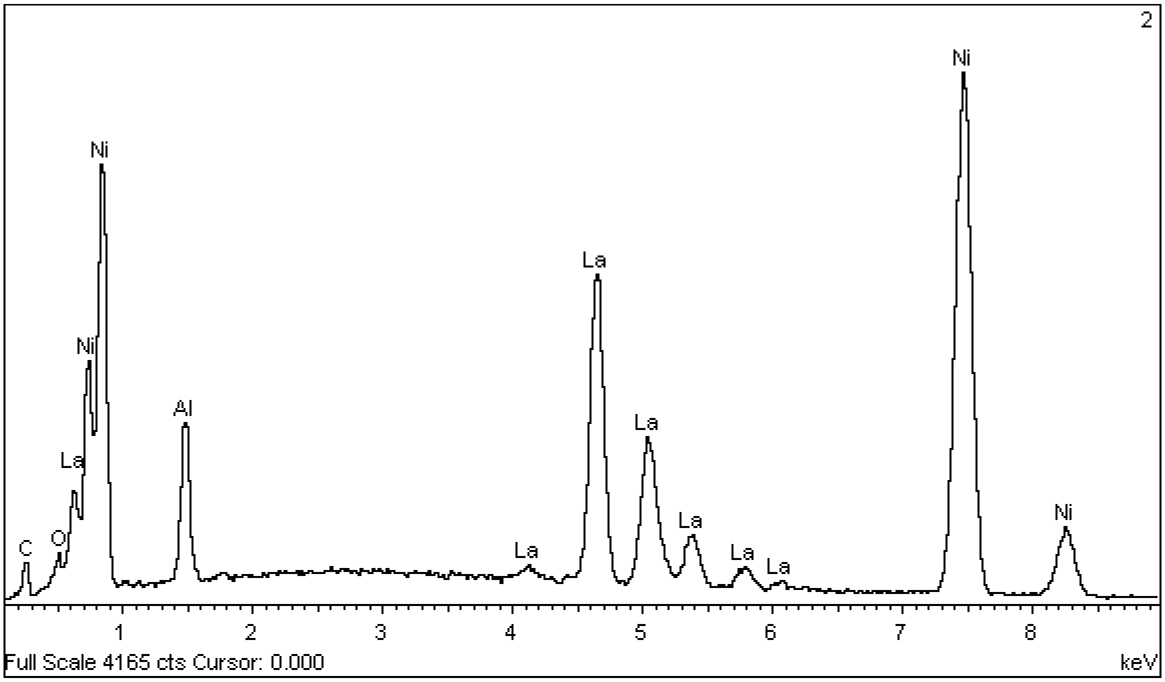

1
O
La
Ni
C
Ni
La
Al
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 9688 cts Cursor: 0.000
keV

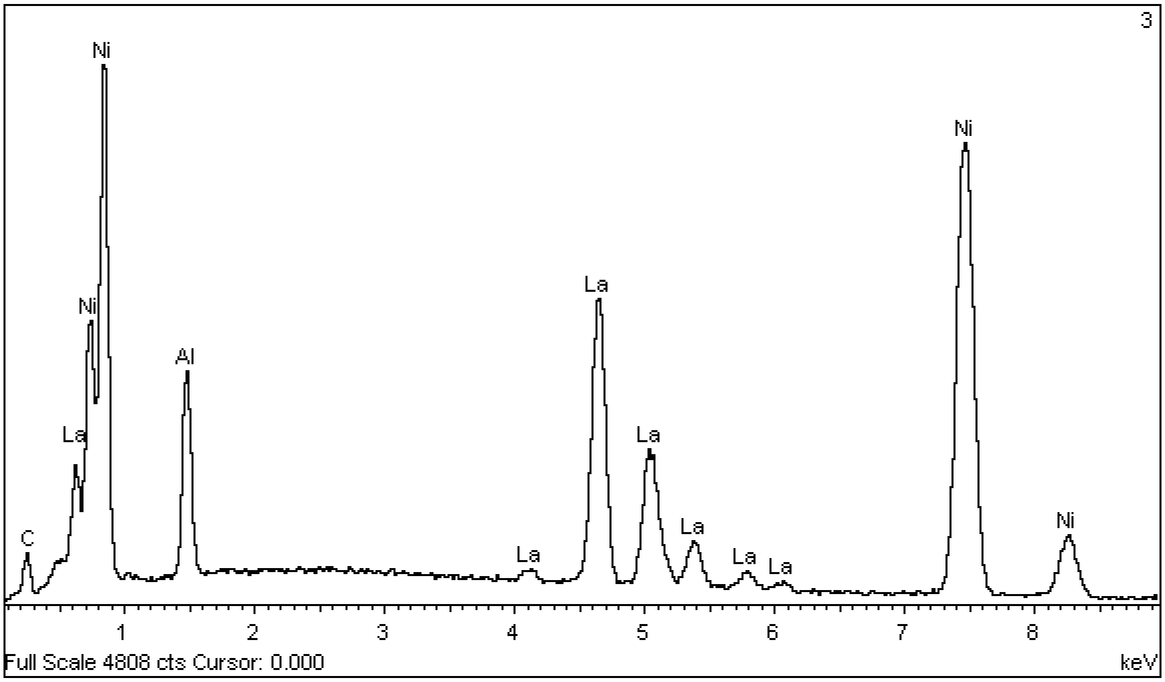

2
Ni
Ni
La
C
O
Al
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
9
Full Scale 8777 cts Cursor: 0.000
keV

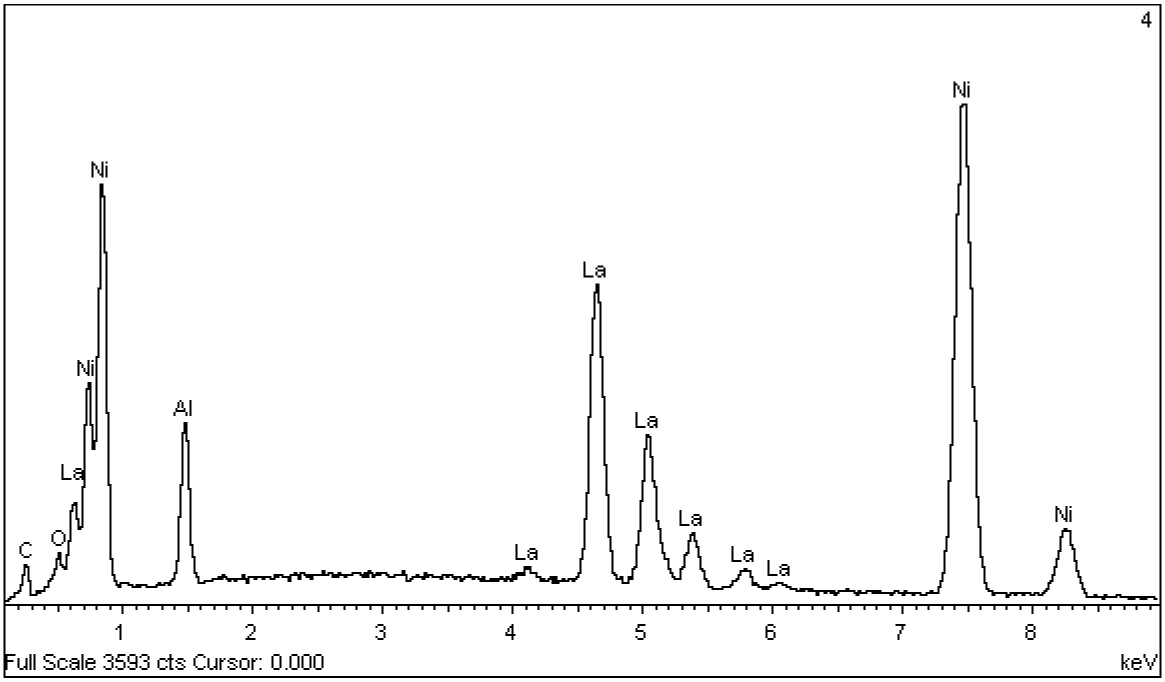


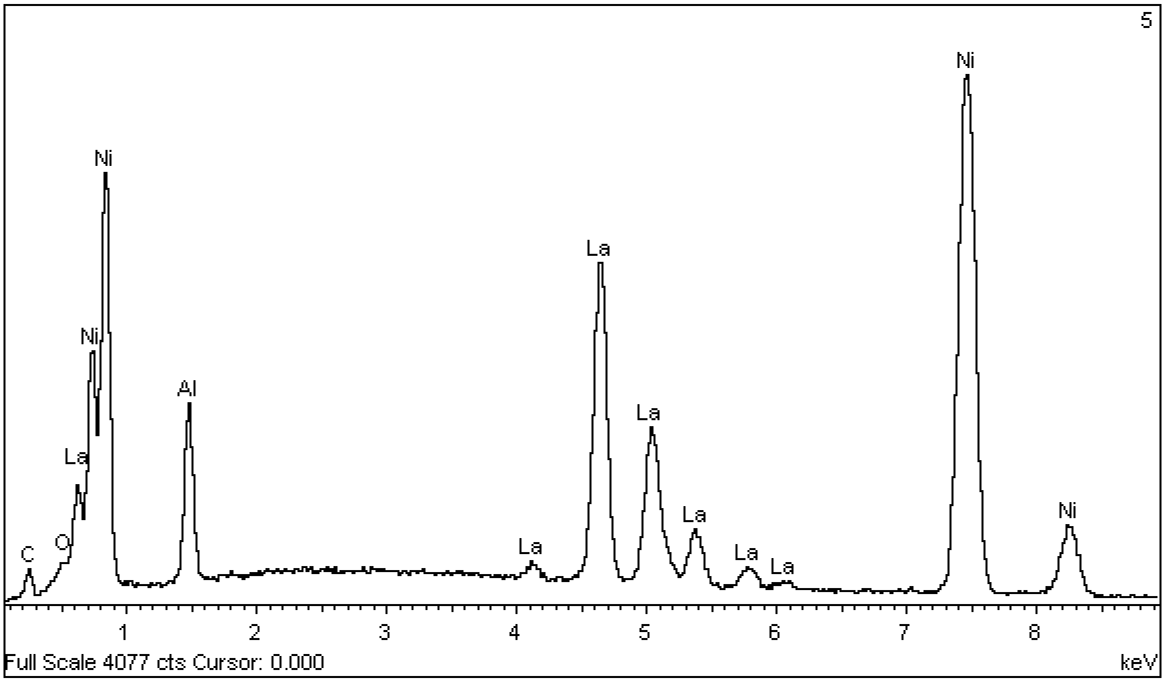
Note: `1` is an area.




2
Ni
Ni
Ni
La
C
O
Al
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 4165 cts Cursor: 0.000
keV


3
Ni
Ni
La
C
Al
La
La
La
La
La
La
Ni
Ni
1
2
3
4
5
6
7
8
Full Scale 4808 cts Cursor: 0.000
keV


4
Ni
Ni
Ni
Al
La
La
La
La
La
La
La
O
C
Ni
1
2
3
4
5
6
7
8
Full Scale 3593 cts Cursor: 0.000
keV


5
Ni
Ni
Ni
Al
La
La
La
La
La
La
La
O
C
Ni
1
2
3
4
5
6
7
8
Full Scale 4077 cts Cursor: 0.000
keV