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Initial Assessment of Physical and Mechanical Properties of Clad/Base Metal Systems

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OAK RIDGE NATIONAL LABORATORY
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Materials Science and Technology Division

INITIAL ASSESSMENT OF PHYSICAL AND MECHANICAL PROPERTIES OF CLAD/BASE METAL SYSTEMS

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September 7, 2018

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ACRONYMS

ART	Advanced Reactor Technologies
ASME	The American Society of Mechanical Engineers
DOE	Department of Energy
ORNL	Oak Ridge National Laboratory
MSR	Molten Salt Reactors

ACKNOWLEDGMENT

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ABSTRACT

In support of the development of new design and construction rules and analysis methods for cladded structure for use under the anticipated service lifetime and cyclic service conditions in Molten Salt Reactors (MSR), research work has been initiated at ORNL for assessment of physical, metallurgical and mechanical properties of clad/base metal systems. This report summarizes the results for the nickel cladded SS316H system in FY18.

This report fulfills the FY18 milestone M4NT-18OR0705020616-“Complete the initial assessment of physical and mechanical properties of clad/base metal systems” under the ORNL work package NT-18OR07050206- “Clad-Base Metal Metallurgical Interaction - ORNL”.

1. INTRODUCTION

The key areas of the Advanced Materials Development (AMD) program for the Molten Salt Reactors (MSR) Campaign are driven by the materials needs due to the significant corrosive effects of molten salts. MSR designs are often limited by the availability of structural materials that have adequate elevated temperature strengths and corrosion resistance to molten salts. Coolant boundary materials per current ASME Section III Division 5 are limited and may not be considered optimum for corrosion resistance with respect to molten salts. Approval of new materials in ASME Section III Division 5 for MSR applications will require comprehensive and very long-term test data.

Integral cladding of corrosion resistant materials on existing Division 5 Class A materials by weld overlay or other cladding manufacturing processes could therefore be an attractive alternative. Cladding is widely used as a design, engineering and manufacturing practice for corrosion protecting and other purposes in many industry applications such as petrochemical refinery systems, and light water nuclear reactors per ASME or other relevant governing codes and standards. Testing requirements of cladding alloys could be less demanding than "load bearing" base metals. Hence, such an option could shorten the development and deployment timelines for MSR. However, the unique MSR operating environments (corrosive molten salts, higher operating temperature, and neutron irradiation damage) would require a thorough evaluation of compatibility of the cladding materials both for corrosion protection performance for MSR and compatibility between the cladding material and the load bearing base structure materials for the anticipated services. This research focuses on the latter, including the microstructure/metallurgical compatibility, the microstructure stability at the cladding/bonding interface, the mechanical property and performance compatibility, and the cladding manufacturability based on the candidate cladding materials and base materials and availability and choice of cladding manufacturing processes. The research will support the development of new design and construction rules and analysis methods for cladded structure for the anticipated service lifetime and cyclic service conditions in MSR.

In this fiscal year (FY2108), research work has been initiated at ORNL for assessment of physical, metallurgical, and mechanical aspects of clad/base metal systems. Two types of clad/metal systems are planned, i.e., compliant clad with nickel as the representative clad material and the elastic clad with molybdenum as the representative clad material. This report summarizes the results for the nickel cladded SS316H system, primarily on progress on the manufacturability by arc weld cladding process, quality of cladding, and microstructure changes in cladding layer and substrate. Additional mechanical testing is on-going and will be reported when the tests are completed.

2. MATERIALS AND METHODS

2.1 MATERIALS

Recent survey identified pure nickel, Hastelloy N (or modified Hastelloy N), molybdenum, tungsten, as a potential corrosion protection clad materials of MSR. Current ASME Section III Division 5 construction materials that may be suitable as structural materials for MSR include SS316H, Alloy 80H, Alloy 617. Alloy 709 is another potential structural material based on its high temperature mechanical properties, but Alloy 709 is not yet code qualified. The survey also suggested that these different cladding alloys would require different cladding technologies to bond onto the above structure materials.

Our initial assessment in FY2018 was focused on cladding of pure nickel on stainless steel 316H. The primary goals are two folds: (1) to determine suitable cladding manufacturing processes to successfully bond/clad pure nickel onto SS316H substrate, and (2) to investigate the metallurgical and microstructural changes in the clad layers resulting from the cladding process, as well as the mechanical properties in the clad and the bonding interface.

The SS316H base metal material (Heat No. 296596) used in this report was manufactured by company SIJ Acroni, Slovenija, EU, in hot-rolled plate form with thickness of 12.7mm (or 0.5-inch). The as-received plate was in solution annealed condition. The chemical compositions of this plate are listed in Table 1. As shown in the table, while meeting ASME SA-240 for SS316H grade, it is noted that this particular heat of SS316H is slightly lower in nitrogen than the new requirement of 0.05% per ASME BVPC.III.5. Also, aluminum content was not listed in the material certification.

Table 1. Chemical compositions of the SS316H base metal (heat 296596), wt.%

C	Si	Mn	P	S	Cr	Ni	Cu	Mo	Ti	Nb	N	Co	Fe
0.041	0.329	1.536	0.036	0.001	16.61	10.26	0.392	2.006	0.005	0.005	0.0315	0.152	Bal.

The microstructure of base metal SS316H steel is shown in Fig. 1. Energy-dispersive X-ray Spectroscopy (EDS) was used to determine the chemical composition of the base metal and the changes in the cladding layers. The circled region in Fig. 1 was used for the EDS measurement and the EDS analysis results of the weight percentage of Fe, Cr and Ni in the base metal are listed in Table 2. The EDS results are very close to the material certification in consideration of the measurement uncertainties. Therefore, it is concluded that the EDS would be a reasonable method to determine the chemical composition changes in the cladding layers of the nickel/SS316H system.

Commercial nickel filler metal wire, ERNi-1 was used for the cladding study. ERNi-1 is a common Ni weld filler metal for welding of commercially pure nickel (Nickel 200 and 201), and also used for dissimilar welding applications including joining commercially Nickel 200 and 201 to stainless steels. The ERNi-1 weld wire used in this study was from Special Metals Welding Products Company (Product Name: Nickle Filler Metal 61 and Heat No. N083WH). The weld wire has a dimeter of 1.143mm (or 0.045-in). The chemical compositions of the weld wire are listed in Table 3, meeting ASME Section II Part C SFA-5.14 2015 Edition and ANSI AWS A5.14/A5.14M:2011. The relatively high Ti in the filler metal is to react with carbon in SS316H base metal during weld cladding to maintain a low level or free carbon for Nickel 200 and 201 clad.

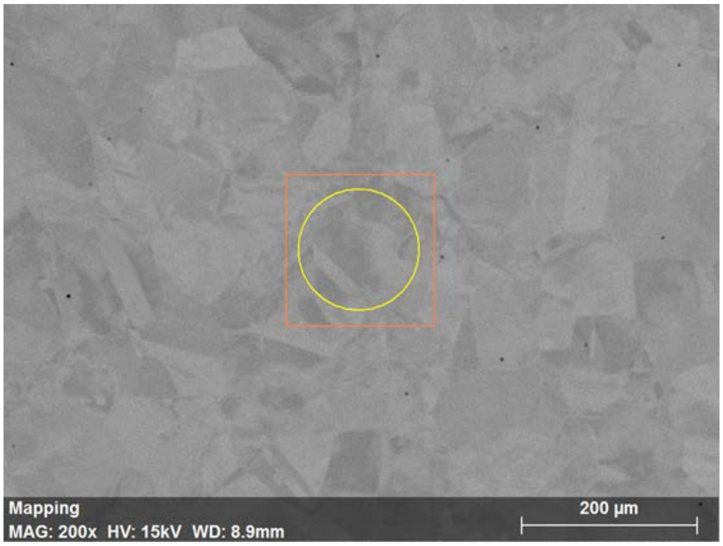


Fig. 1. SEM image of the SS316H base metal

Table 2. EDS measurements of the base metal SS316H

Element	Composition, wt.%	Error %
Iron	67.71	2.00
Cr	16.19	0.50
Ni	9.55	0.30

Table 3. Chemical compositions of the ERNi-1 nickel filer metal (heat N083WH), wt.%

C	Si	Mn	P	S	Al	Ti	Ni	Cu	Fe	Others
0.006	0.270	0.726	0.001	0.004	0.152	2.746	95.670	0.017	0.287	<0.50

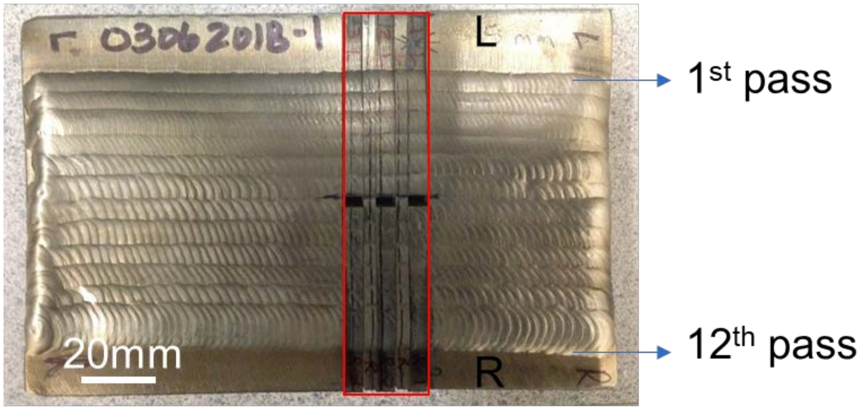
2.2 WELD OVERLAY CLADDING

The nickel filler metal ERNi-1 was cladded onto SS316H base metal using the Gas Tungsten Arc Welding (GTAW) process, in flat-1G position. Semi-automatic welding with argon shielding gas was used to produce the weld overlay/clad on the SS316H plate. Nominal welding current and voltage were 155A and 10V respectively.

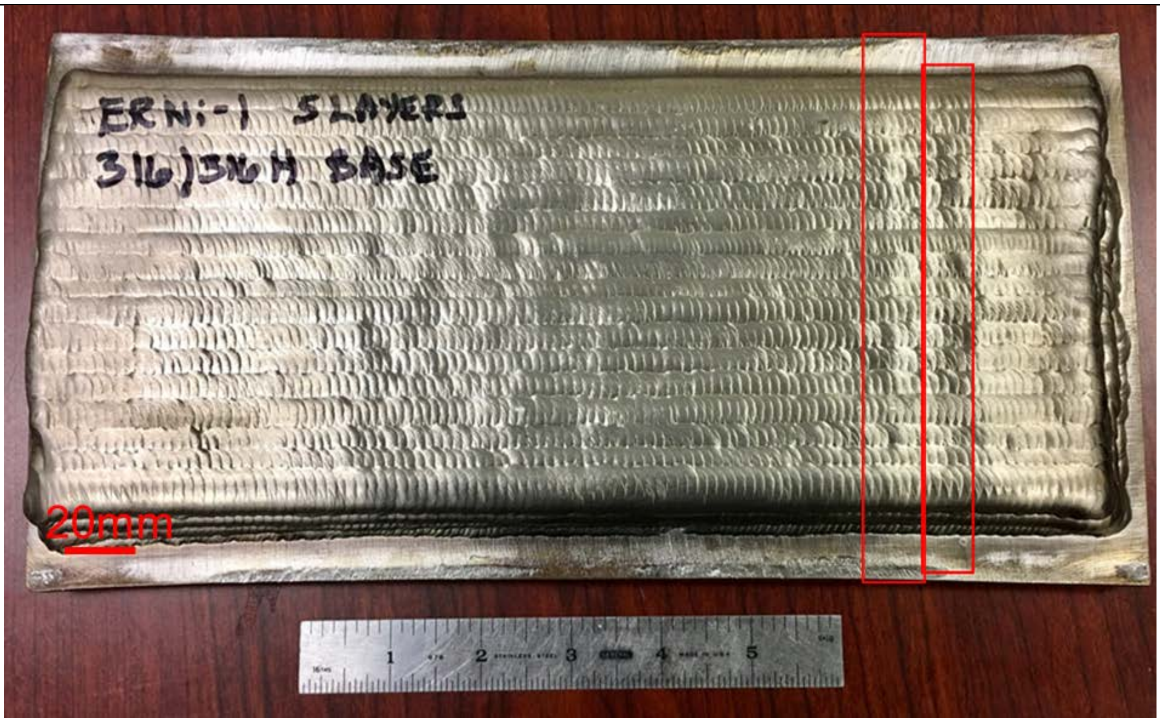
Both single layer weld overlay and multi-layer overlay were studied in FY2018. A 6-inch long- 4-inch wide and 0.5-inch thick SS316H base metal pad was used in the initial single layer cladding study. The single layer nickel clad was made with 12 weld passes with 50% overlap. The thickness of the single layer clad was approximately 3mm. The five-layer cladding was built up on a 12-inch long – 6-inch wide and 0.5-inch thick SS316H pad, with same nominal welding parameters. It resulted in the total clad thickness about 13mm. A picture of the two clad pads are shown in Fig. 2. The red boxed regions are where specimens were machined for bending tests and metallurgical characterization.

The fabricated clad pads went through ASME SEC IX weld clad qualification specifications, including die penetrant inspection, X-ray radiography non-destructive inspection, and side bend testing. Side bend specimens were machined out of the clad pads perpendicular to the welding direction. Side bend test were conducted for both single layer and multi-layer nickel clad. The nominal thickness of the bending specimens was 9.5mm (or 3/8-in).

Chemistry and microstructure characterizations were performed using Quantax70 Energy-dispersive X-ray spectroscopy (EDS) and TM3030plus Scanning Electron Microscope (SEM).



(a) Single layer Nickel clad on SS316H



(b) 5-layer Nickel clad on SS316H

Fig. 2. Single layer and 5-layer Nickel clad pad on SS316H.

3. ASSESSMENT OF NICKEL CLAD ON SS316H BASE METAL

3.1 QUALIFICATION TEST RESULTS OF NICKEL CLADED SS316H

Nondestructive Testing (NDE) was performed on the single layer and multi-layer Nickel cladded SS316. Both clad pads do not show relevant indications of defects through dye penetrant inspection. X-ray radiographic inspection image of the 5-layer Nickel clad shown in Fig. 2(b) is presented in Fig. 3, and the inspection shows that it passes ASME IX QW 191.1.2.2 qualification requirements.

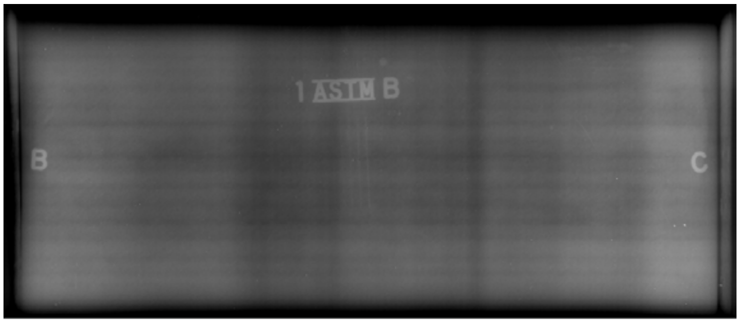


Fig. 3. X-ray inspection of the 5-layer Nickel clad on SS316H

Wrap around bend tests were performed on four specimens from the multi-layer Nickel clad, and plunge bend tests were performed on four specimens from the single layer clad. Pictures of all the tested specimens are show in Fig. 4. The red dash lines in Fig. 4 identify the location of the boundary between the Nickel clad and the SS316 base metal. No visible cracks were found on the four single layer clad specimens after bending tests. Some microcracks were formed after bending tests of the multi-layer Nickel clad but the cracks are less than 1.5mm (or 1/16-in), the code allowable size. Both single layer Nickel clad and multi-layer Nickel clad have therefore passed the ASME SEC IX qualification evaluation.

Fig. 4. Side-bend test results of the Nickel clad on SS316H

3.2 MICROSTRUCTURE AND COMPOSITION ANALYSIS OF SINGLE-LAYER CLADDING PAD

Metallurgical specimens were machined from the single layer Nickel clad pad and prepared for microstructure and composition analysis. The entire cross-section area was first examined under optical microscope, and no cracks or visible defects were observed. The optical images of the cross-section of the single layer Nickel clad are shown in Fig. 5.

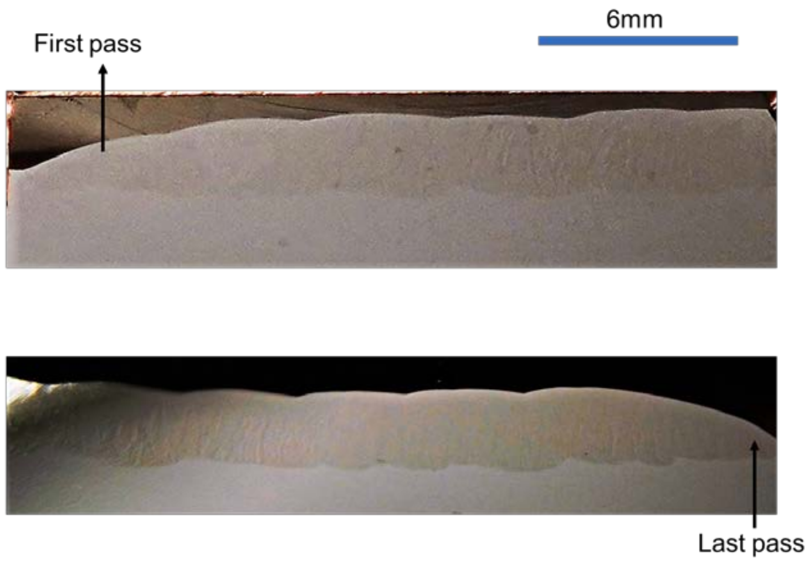


Fig. 5. Optical images of polished cross-section of the single-layer Nickel clad pad on SS316H

SEM microstructure images of the weld pass 1 to 6 and pass 7 to 12 for the single layer Nickel clad are shown in Fig. 6 and Fig. 7. Small voids about 50µm in size were found at this magnification and the largest voids was about 200µm in size as shown on pass 4 and pass 11. These voids are small in size, cannot be revealed through X-ray inspection, and are acceptable per ASME Section IX for weld clad qualification requirements.

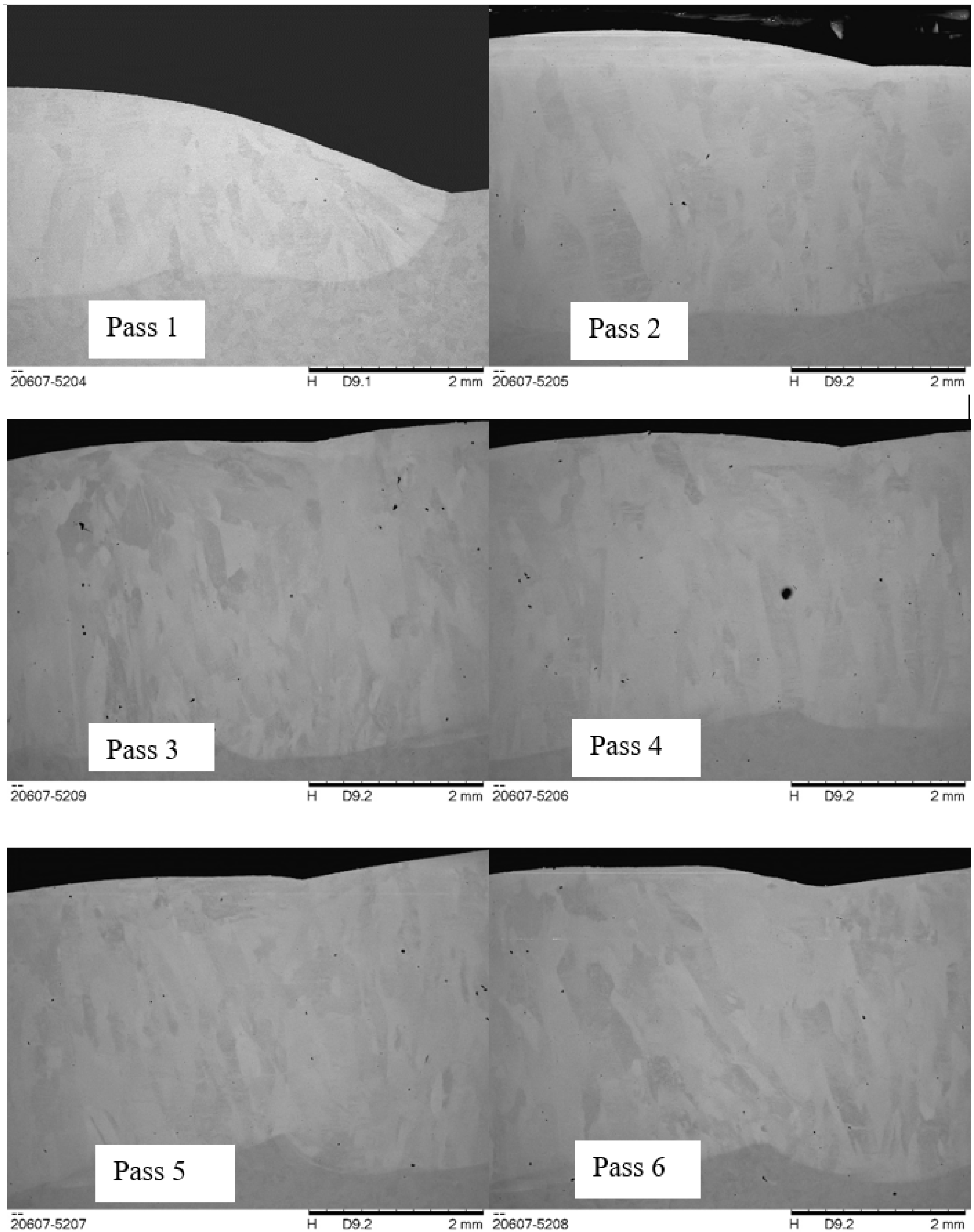


Fig. 6. SEM images of cladding at different area from pass 1 to pass 6.

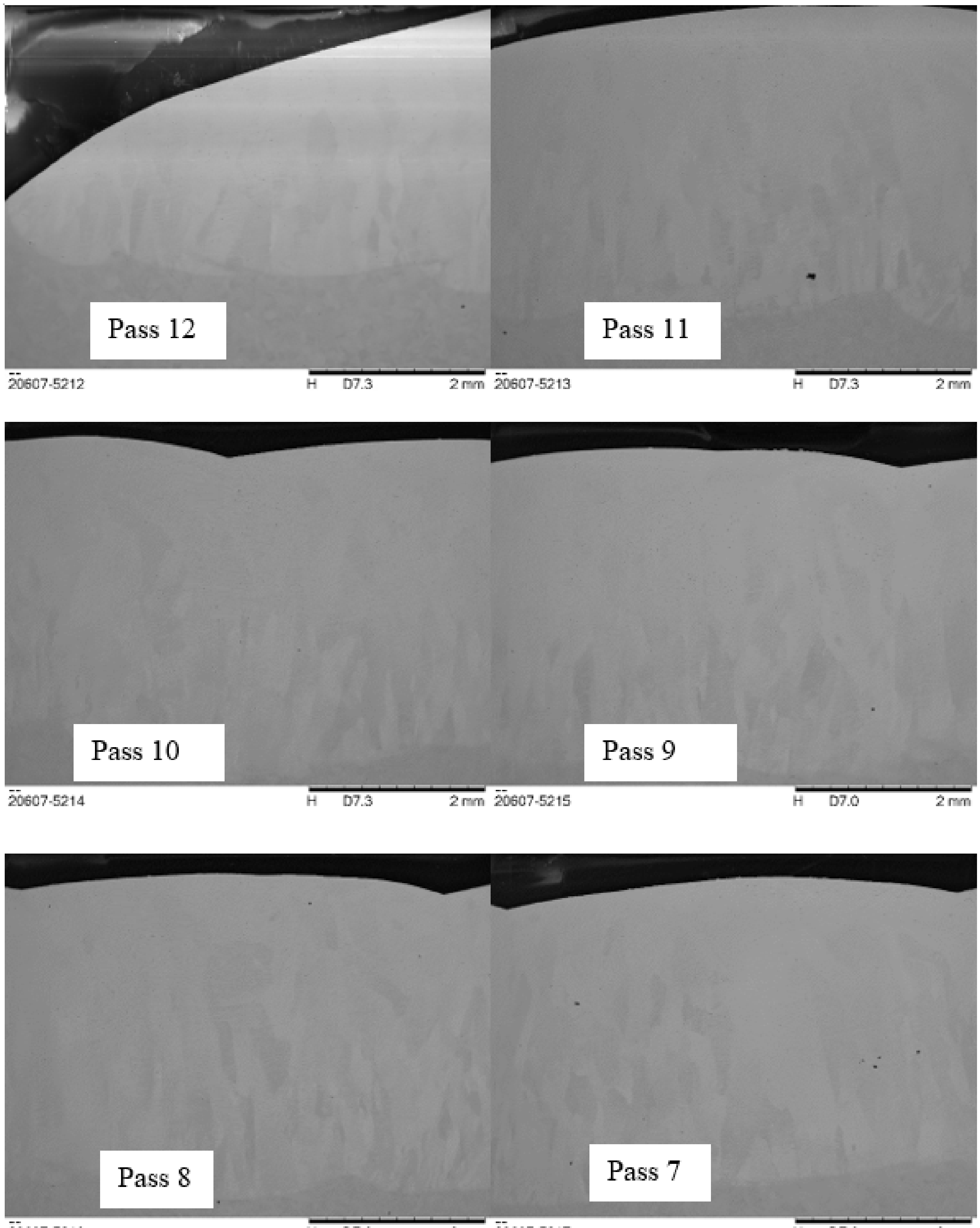


Fig. 7. SEM images of cladding at different area from pass 7 to pass 12.

For all the 12 clad passes, chemical compositions were analyzed at three locations with respect to clad/ base metal interface and these three locations are illustrated with yellow and red circles of the cross-sectional area in Fig. 8. The two locations near the clad/base metal interface boundary, one above and the other below the interface had a 200μm diameter probe size (shown as the two yellow circles). The chemical compositions were also analyzed at the mid-thickness of the clad with a 300μm diameter probe size (shown as the red circle).

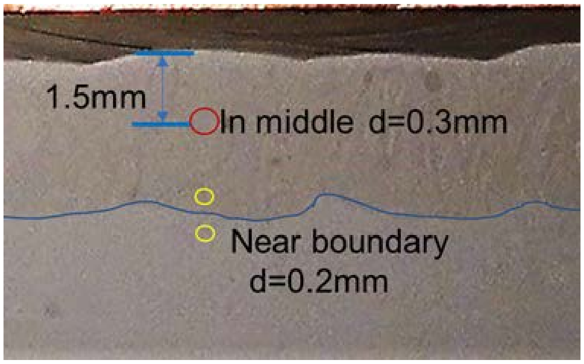


Fig. 8. Probed locations for EDS chemical composition analysis

The results of the EDS analysis on pass 1, pass 2, pass 3, pass 4, pass 6 and pass 11 of the single layer clad are presented in Fig. 9 to Fig. 14, respectively. EDS line profiles (shown with the yellow lines in image (a) and (c) in each figure) across the clad/base metal boundary are presented in image (d) and (e) for each weld passes in these figures. The line profiles are scanned from the top of the clad layer into the base metal. The line profile can qualitatively reveal the chemical composition changes. An element map across the clad/base metal boundary is shown as image (b) for each weld pass. The element mapping area is identified by the red-box in the image (a) for each weld pass.

The changes of chemical composition Fe, Ni and Cr as a function of passes are summarized in Fig. 15. The dilution effect (chemistry mixing of the weld wire and the molten base metal) was evident in the single layer clad (under the arc weld cladding condition used). First, within each clad pass, there was a transition of Fe, Ni, Cr concentration cross the fusion boundary and continuous change inside the clad layer. Second, the Fe, Ni, and Cr concentration changed from one pass to the next but become homogenized within a pass and reached a state of "saturation" after 6 passes, where only ~80wt.% Ni in the cladding layer vs 95.6wt.% Ni in the weld wire. The results from this single layer clad study has granted the need for further research on multi-layer cladding for this Nickel/SS316H system.

Another important observation is that that the Cr content is reduced significantly after just a single layer cladding. It was reduced to about 6.6% in the first pass, and further reduced to about 2.2%. The Cr distribution was also relatively homogeneous through the thickness in the Ni cladding layer. For comparison, the Cr content is nominally 16.5% of 316H SS. Studies in the past suggested that the preferential dissolution of Chromium from both Nickel-based alloys and austenitic stainless steels is a contributing factor for corrosion in FLiBE. It was one of the reasons to limit the Cr to 7 wt.% in Hastelloy N which is widely regarded to have excellent corrosion resistance in molten salt environment. Our study suggests potential effectiveness of a single layer of Ni-201 cladding for corrosion prevention, which would certainly require further experimental corrosion testing for verification.

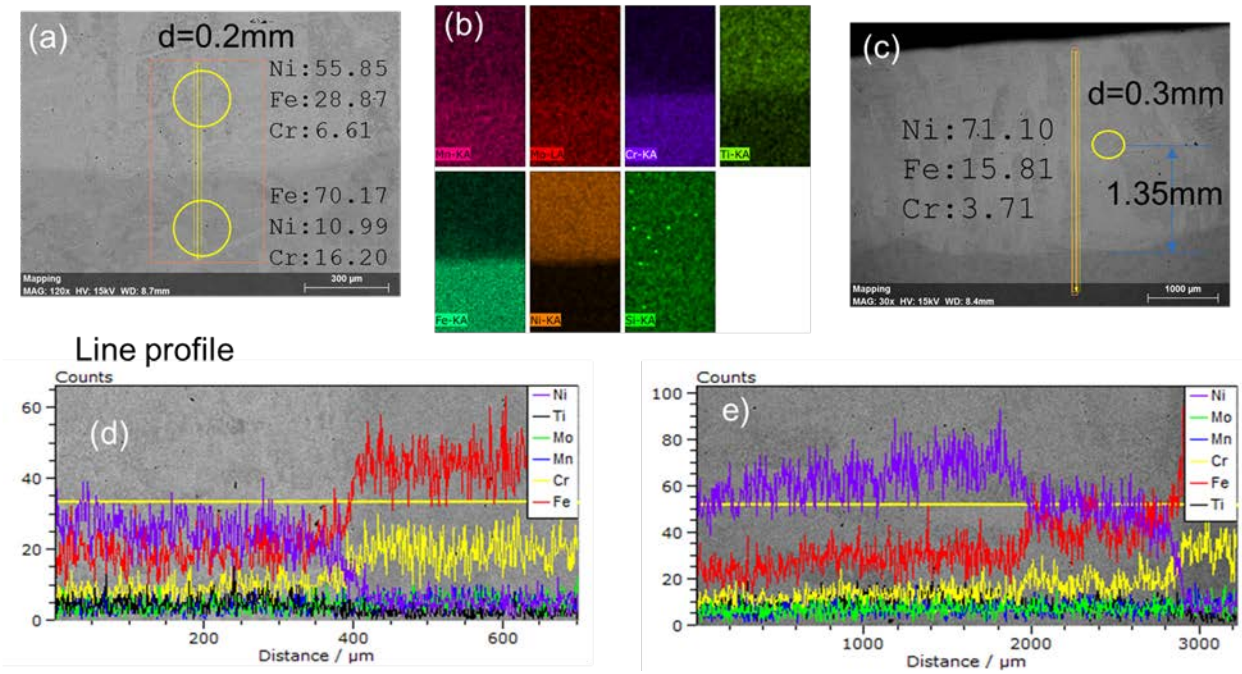


Fig. 9. Analysis of the single layer Nickel clad on S316-weld pass 1. Image and EDS chemical composition near the cladding boundary (a), chemical composition mapping (b), chemical composition away from the clad/base metal boundary (c), EDS line profile (d) for the yellow line in (a) and EDS line profile (e) for the yellow line in (c).

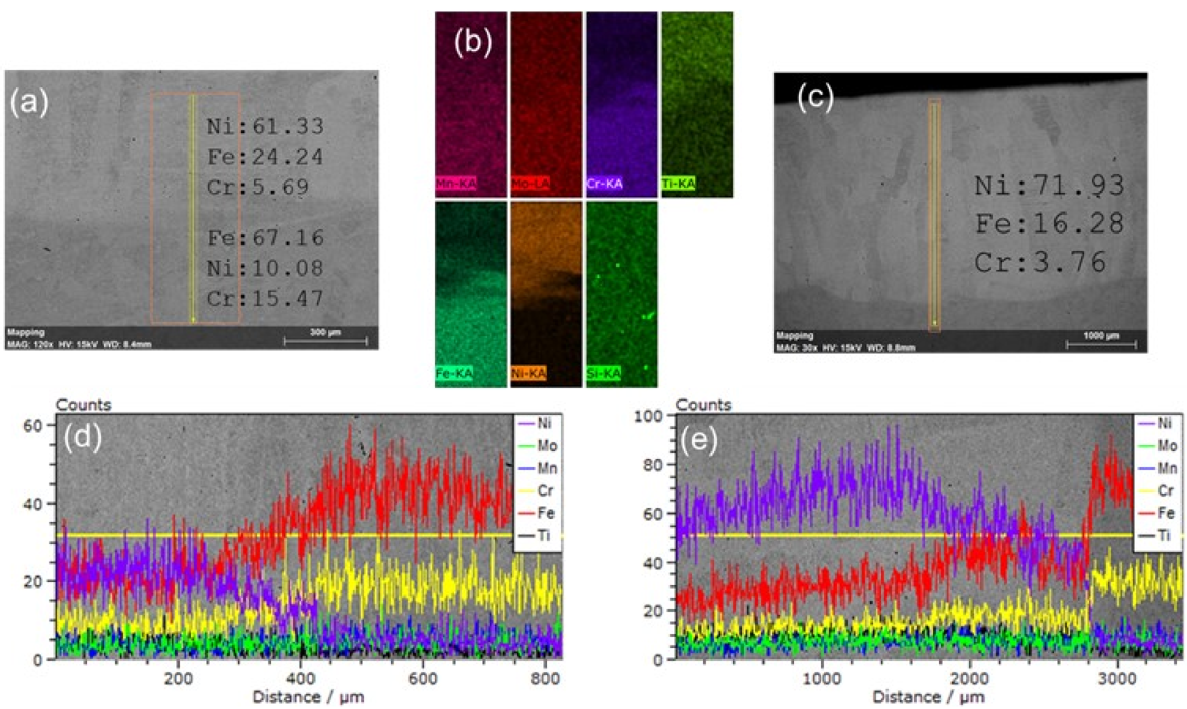


Fig. 10. Analysis of the single layer Nickel clad on S316-weld pass 2. Image and EDS chemical composition near the cladding boundary (a), chemical composition mapping (b), chemical composition away from the clad/base metal boundary (c), EDS line profile (d) for the yellow line in (a) and EDS line profile (e) for the yellow line in (c).

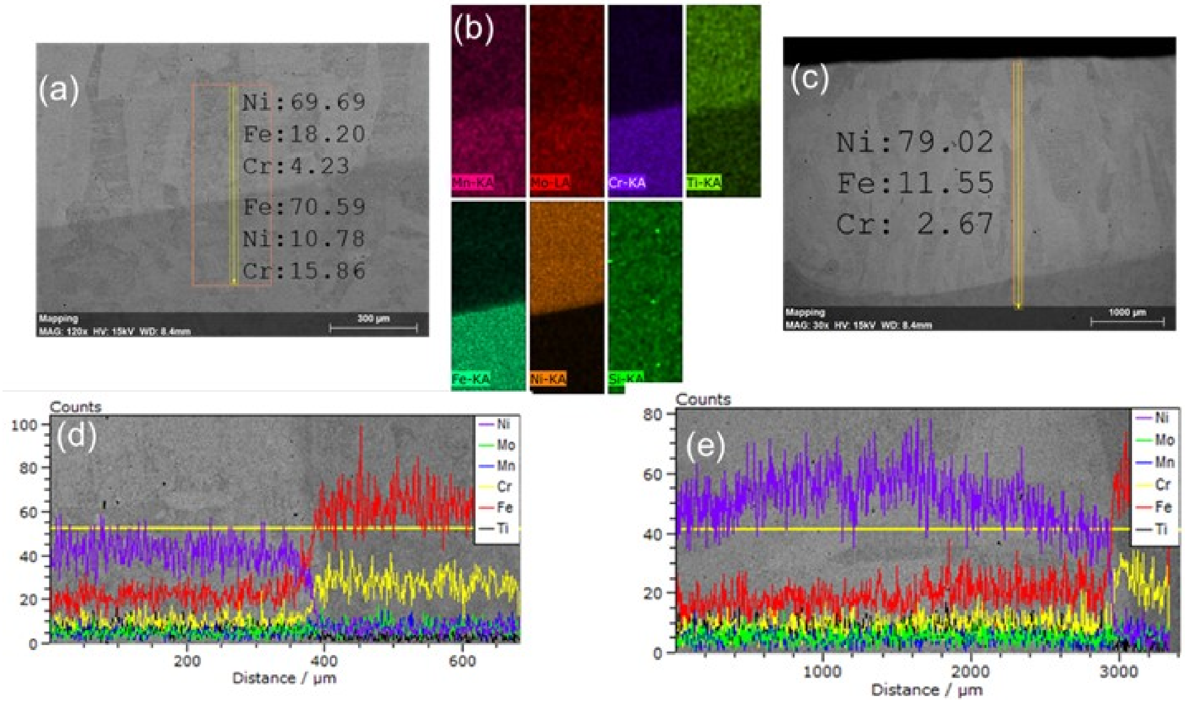


Fig. 11. Analysis of the single layer Nickel clad on S316-weld pass 3. Image and EDS chemical composition near the cladding boundary (a), chemical composition mapping (b), chemical composition away from the clad/base metal boundary (c), EDS line profile (d) for the yellow line in (a) and EDS line profile (e) for the yellow line in (c).

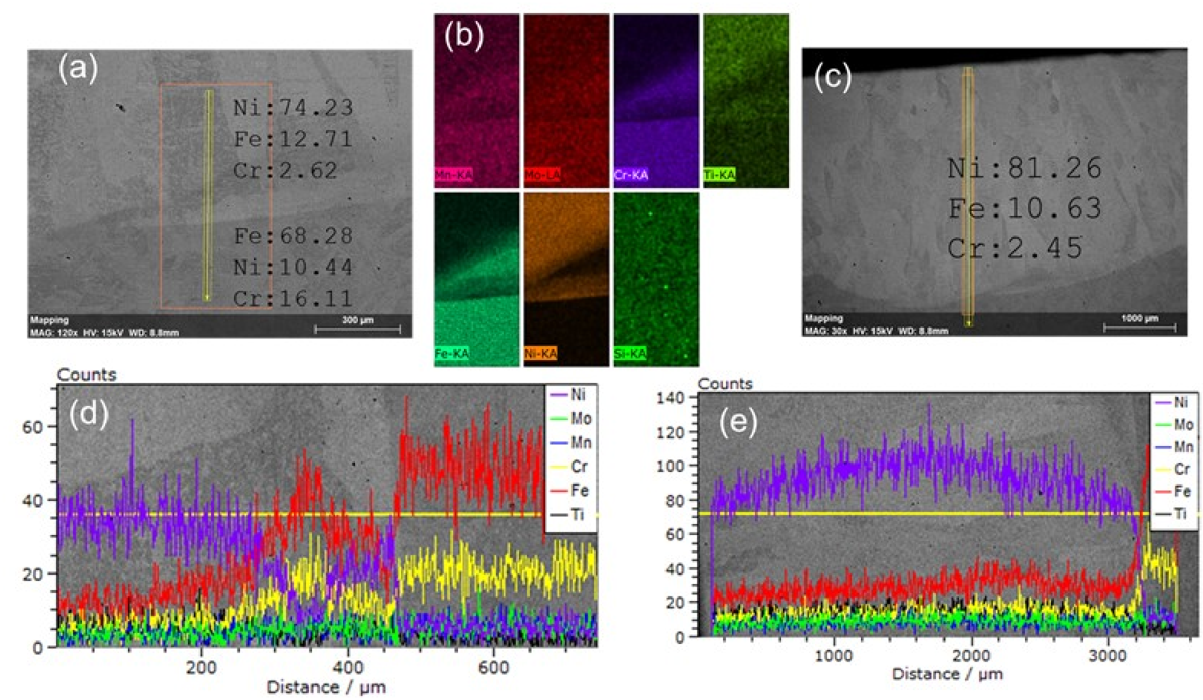


Fig. 12. Analysis of the single layer Nickel clad on S316-weld pass 4. Image and EDS chemical composition near the cladding boundary (a), chemical composition mapping (b), chemical composition away from the clad/base metal boundary (c), EDS line profile (d) for the yellow line in (a) and EDS line profile (e) for the yellow line in (c).

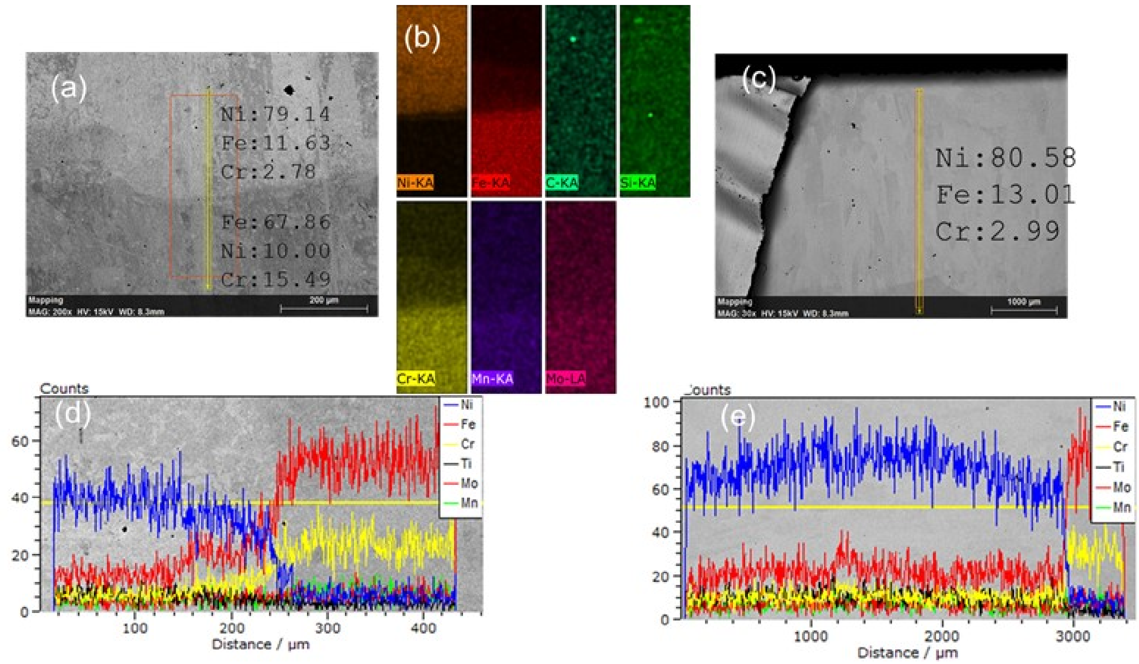


Fig. 13. Analysis of the single layer Nickel clad on S316-weld pass 6. Image and EDS chemical composition near the cladding boundary (a), chemical composition mapping (b), chemical composition away from the clad/base metal boundary (c), EDS line profile (d) for the yellow line in (a) and EDS line profile (e) for the yellow line in (c).

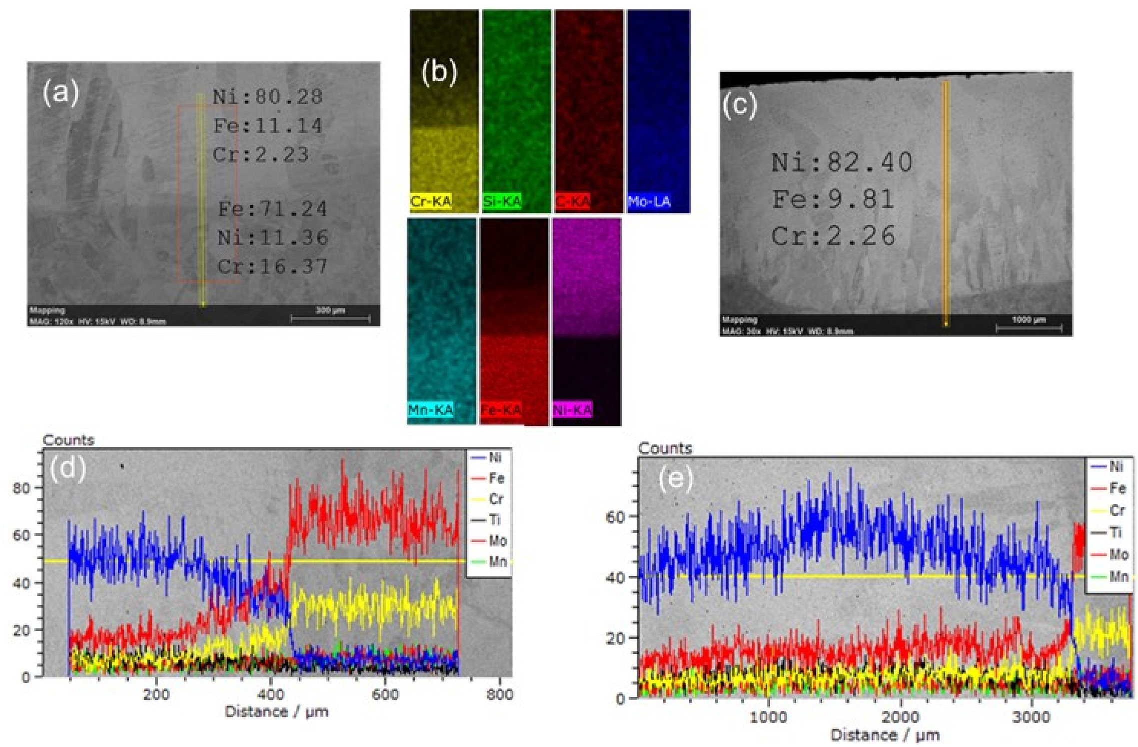


Fig. 14. Analysis of single layer Nickel clad on S316-weld pass 11. Image and EDS chemical composition near the cladding boundary (a), chemical composition mapping (b), chemical composition away from the clad/base metal boundary (c), EDS line profile (d) for the yellow line in (a) and EDS line profile (e) for the yellow line in (c).

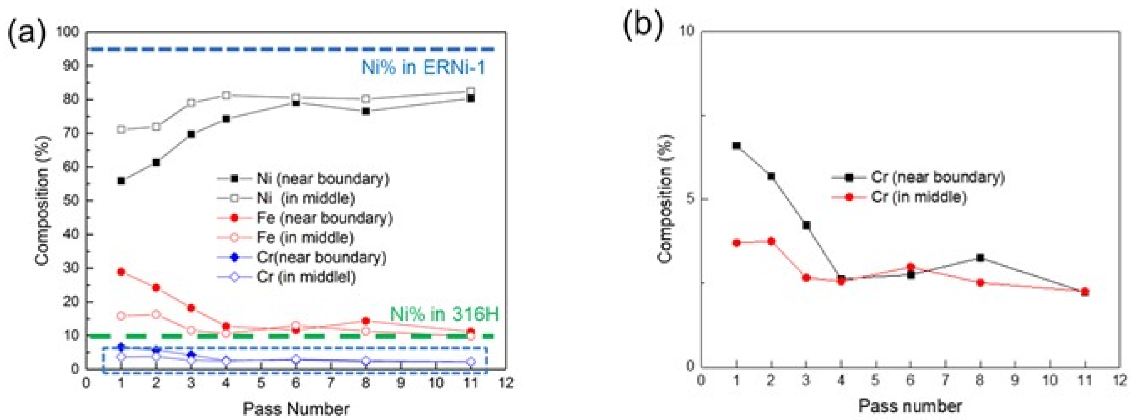


Fig. 15. Composition evolution of Fe, Ni and Cr as a function of weld passes

3.3 MICROSTRUCTURE AND COMPOSITION ANLAYSIS OF MULTI-LAYER CLADDING PAD

A multi-pass and 5-layer nickel clad pad was made on the SS316H base metal (shown in Fig. 2(b)). Optical images of the cross-sectional area of this 5-layer clad are presented in Fig. 16. There were small voids present in the nickel clad, and the largest voids found were approximately 1mm and highlighted in the red circle in Fig. 16. The size of these voids is permitted per ASME IX qualification requirements.

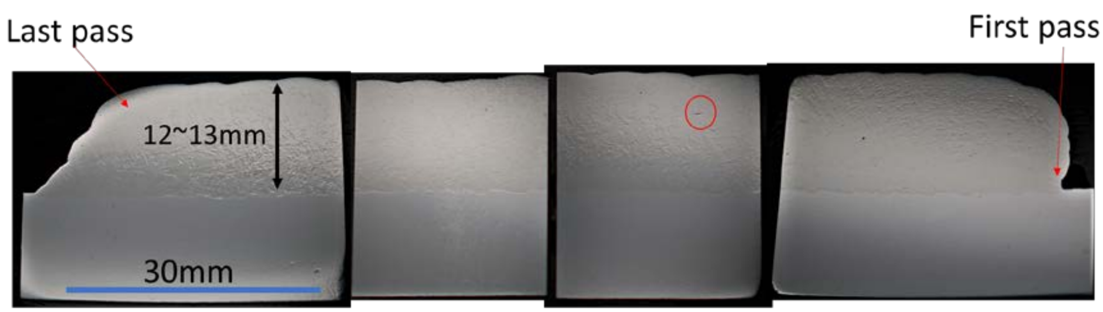


Fig. 16. Optical microscope images of the cross-section of the multi-pass and 5-layer Nickel clad pad on SS316H base metal

Detailed microstructure characterization and chemical composition analysis were performed on the 5-layer Nickel clad and the results are presented for the weld passes 1, 2, 4, 6 and 9 in Fig. 17 to Fig. 21, respectively. Weld pass number 9 is at about the mid-width of the clad pad. The pass number used here represents the weld passes of all the five layers. In these figures, the microstructure and chemical compositions at the Nickel/SS316 boundary are shown in (a) and the element maps of the chosen area across the boundary are shown as (b). The content of Ni, Fe and Cr along the entire clad thickness are plotted in (c). The locations of the chemical composition measurement are identified on the cross-sectional images (d).

The analysis shows that across the clad thickness, the dilutions is significant at the boundary and decreases with additional clad layers. For all the weld passes examined, the Ni content approaches the maximum and equals to the concentration in the weld wire at about 6 to 7 mm thickness from the Ni/SS316H boundary. The Fe and Cr concentration diminish to zero at similar distance. Fig. 22 demonstrated a 2-D map for the Nickel concentration for all the locations measured. The map clearly shows that the Ni content is uniform across the clad pad width and reached to the maximum when the clad thickness is above 6 mm. This suggests that a minimum of three Nickel clad layers are required for this Ni clad/SS316H system to reach desirable clad chemistry.

The above discussions are based on the results from the study in this report. For high temperature MSR applications, additional factors such as aging effect, corrosion resistance and radiation effect must be evaluated. The results in this report provide basic information for such future studies.

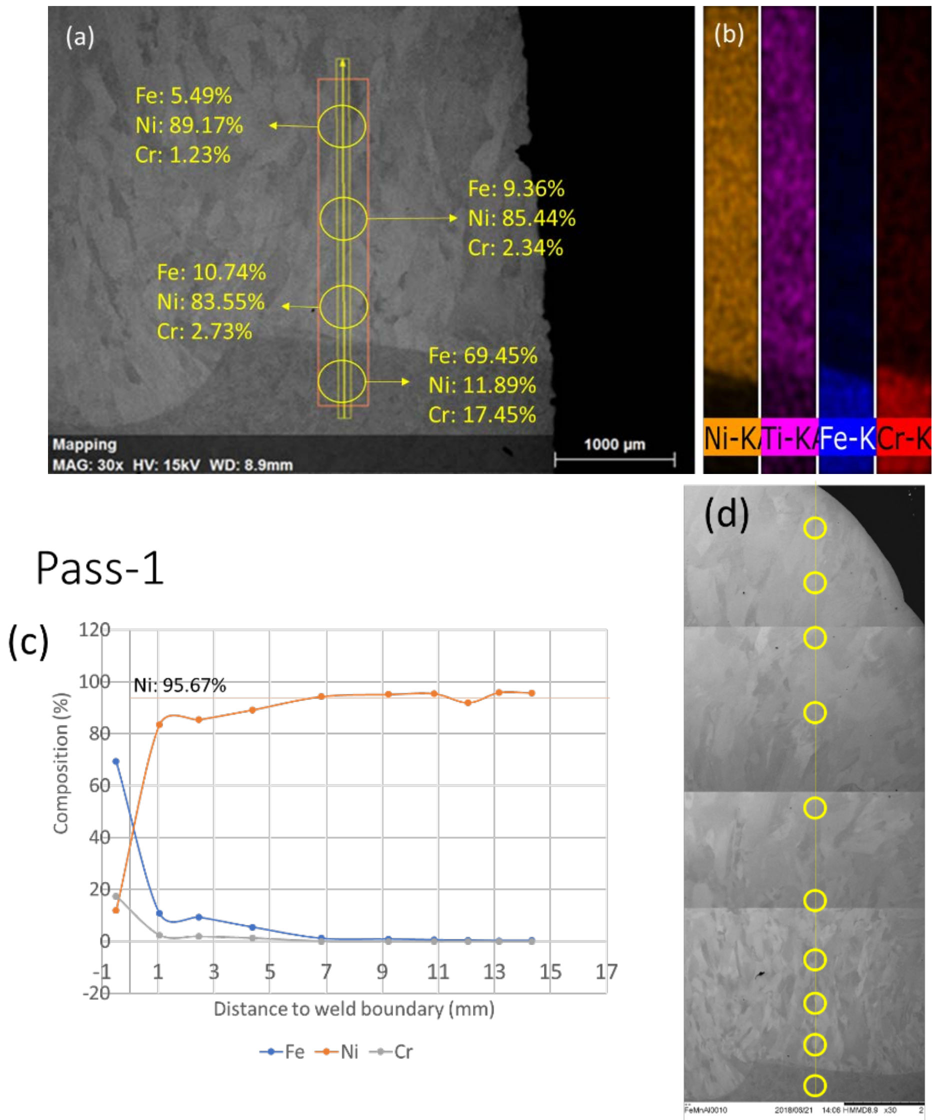


Fig. 17. Analysis of the 5-layer Nickel clad on S316-weld pass 1. (a) composition of Fe, Ni and Cr near the boundary. (b) composition mapping in (a). (c) composition evolution of Fe and Ni as a function of distance to boundary. (d) SEM image and locations of composition detected in (c).

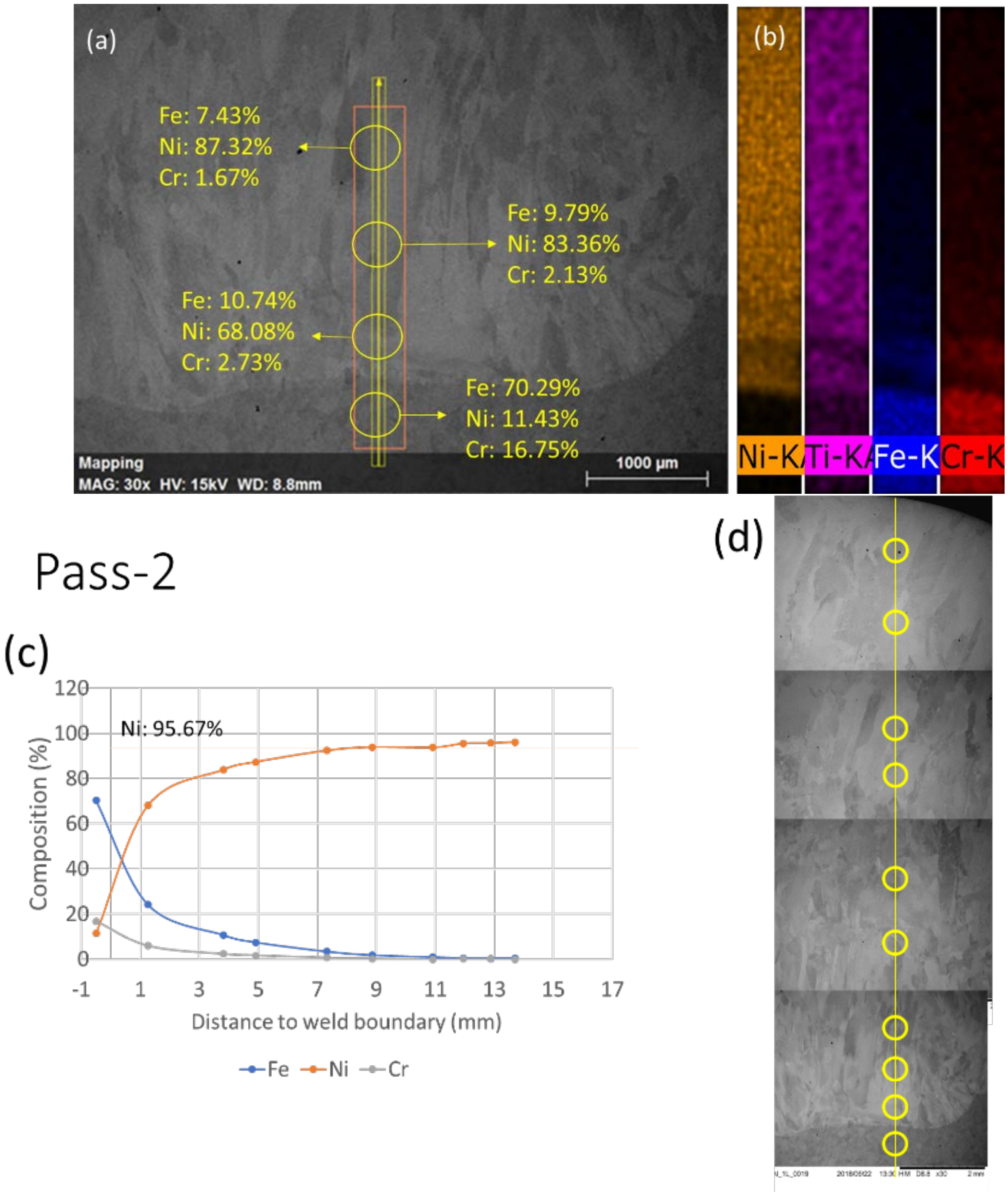


Fig. 18 Analysis of the 5-layer Nickel clad on S316-weld pass 2. (a) composition of Fe, Ni and Cr near the boundary. (b) composition mapping in (a). (c) composition evolution of Fe and Ni as a function of distance to boundary. (d) SEM image and locations of composition detected in (c).

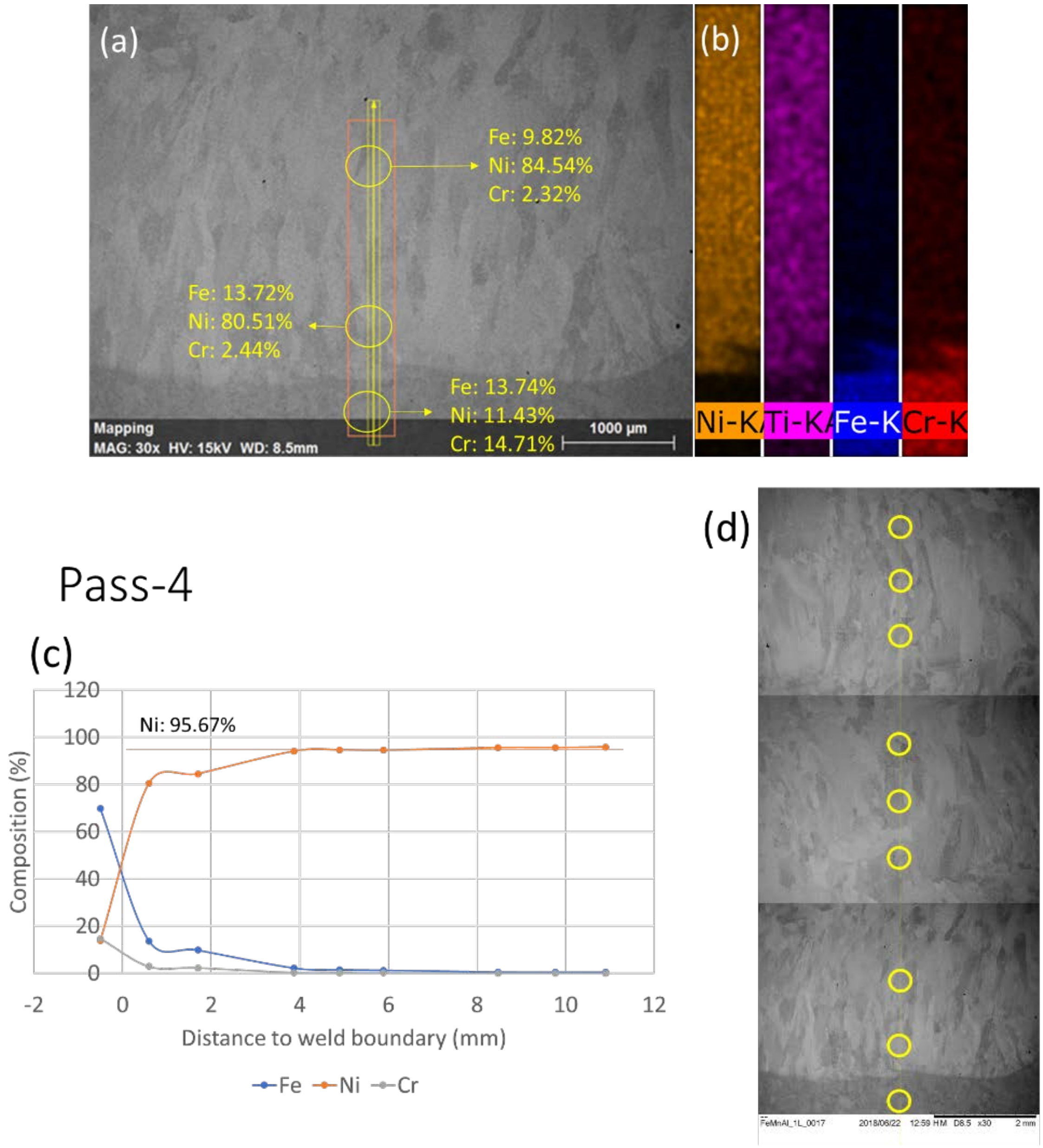


Fig. 19. Analysis of the 5-layer Nickel clad on S316-weld pass 4. (a) composition of Fe, Ni and Cr near the boundary. (b) composition mapping in (a). (c) composition evolution of Fe and Ni as a function of distance to boundary. (d) SEM image and locations of composition detected in (c).

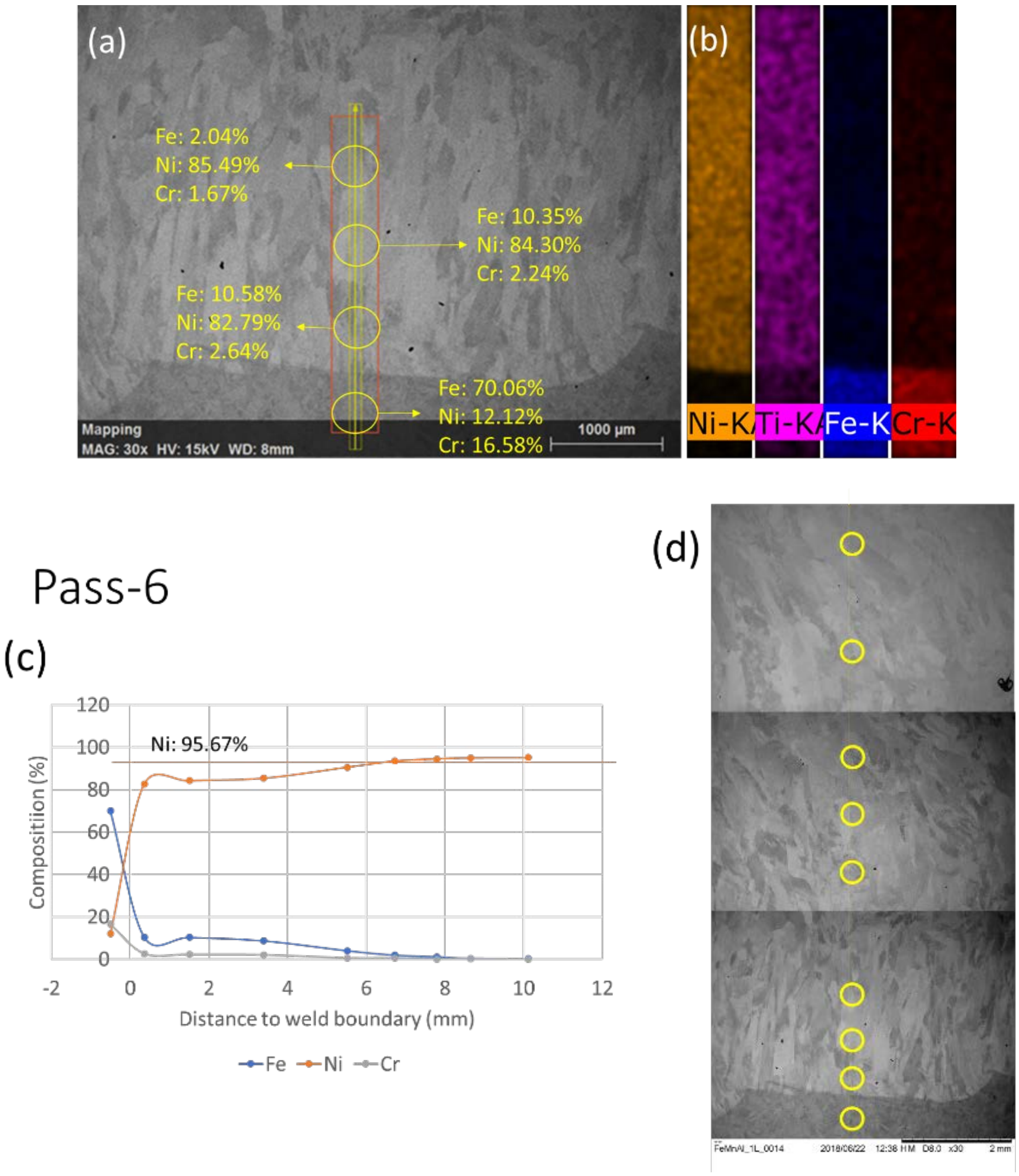


Fig. 20. Analysis of the 5-layer Nickel clad on S316-weld pass 6. (a) composition of Fe, Ni and Cr near the boundary. (b) composition mapping in (a). (c) composition evolution of Fe and Ni as a function of distance to boundary. (d) SEM image and locations of composition detected in (c).

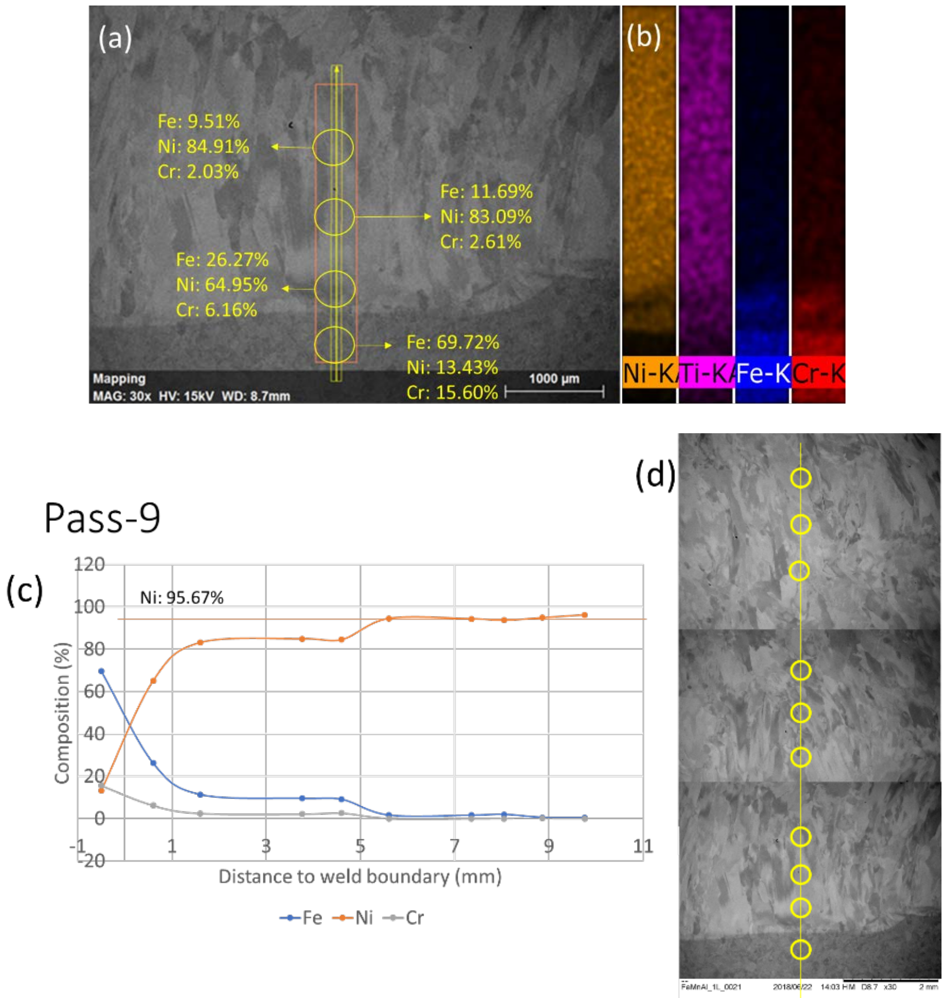


Fig. 21. Analysis of the 5-layer Nickel clad on S316-weld pass 9. (a) composition of Fe, Ni and Cr near the boundary. (b) composition mapping in (a). (c) composition evolution of Fe and Ni as a function of distance to boundary. (d) SEM image and locations of composition detected in (c).

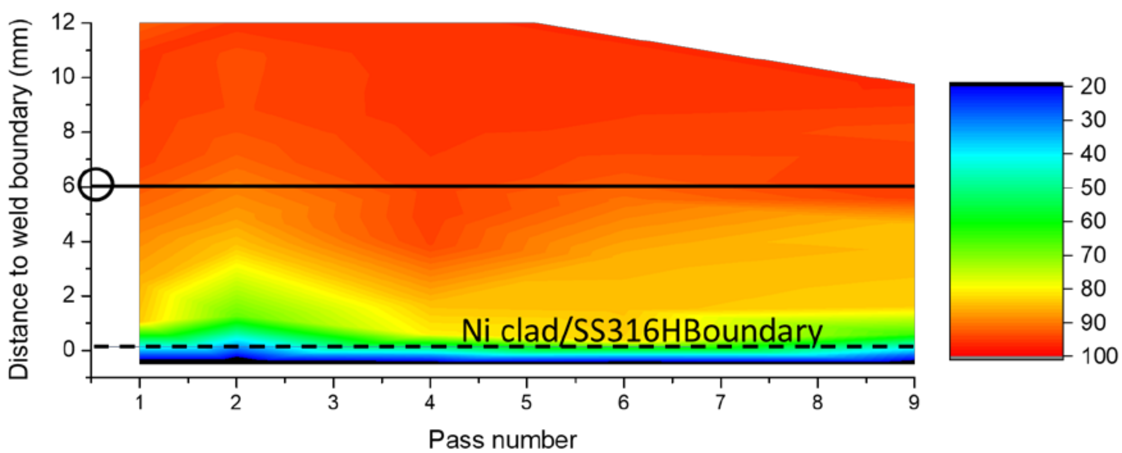


Fig. 22. Composition map of Ni

4. SUMMARY

This report summarizes the initial assessment of the single layer and 5-layer Nickel cladded SS316H system. The study shows that the Nickel cladded SS316H system can be successfully manufactured through a standard arc welding process. Detailed microstructure characterization, clad chemistry analysis and mechanical testing revealed that (1) In a single layer clad, the Nickel content is reduced to about 80 wt.% due to the dilution effect from mixing of ERNi-1 filler metal of 95.6 wt.% and the molten SS316H base metal; and the Cr level is effectively reduced to 2%. (2) A minimum of three layers of Nickel clad are required to minimize the chemical composition dilution. Additional tests such as high temperature aging effects or corrosion resistance are suggested for the application of the Nickel cladded SS316H system as components in the MSR.

This report fulfills the FY18 milestone M4NT-18OR0705020616-“Complete the initial assessment of physical and mechanical properties of clad/base metal systems” under the ORNL work package NT-18OR07050206- “Clad-Base Metal Metallurgical Interaction - ORNL”.

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