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Study on Composition-Induced Microstructural Variation in the Interface Between Co-Based Hardfacing Alloys and IN738 Ni-Based Superalloy

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Alloy mixtures with varying composition of two Co-based hardfacing alloys and IN738 Ni-based super alloy were examined using differential scanning calorimetry. The microstructure of the resulting specimens was then analyzed using optical and scanning electron microscopy and energy dispersive spectroscopy. The microstructural evolution between each hardfacing alloy and the IN738 substrate was documented and the formation of topologically close-packed and/or additional carbide phases was noted. Microstructures that are undesirable for high temperature and stress applications were produced in specimens with a higher degree of mixing (from 25 to 75 wt.% of hardfacing alloy in IN738). The two hardfacing alloys produced markedly different microstructures in the alloy mixtures due to differences in elemental composition. A hardfacing process that minimizes the degree of deposit-substrate mixing will be beneficial to the performance of the coating.

Keywords DSC, hardfacing, IN738, microstructure, Stellite 694, Tribaloy T-800

1. Introduction

Gas turbine engine operating efficiency is highly dependent on the material capabilities of components in the hot sections of the engine. Specialized materials and manufacturing methods are continually developed for parts such as turbine blades that experience extreme operating environments. These components/materials are designed to withstand high stresses and temperatures with little effect on the integrity of the blade. To impart all these capabilities, superalloys have been developed that contain various alloying elements such as Ta, Nb, Zr, and Re, and in combination with highly controlled and sophisticated processing methods, it becomes very costly to manufacture, and hence to replace, turbine blades. Surfacing techniques can be used to apply protective coatings to new turbine blades for added wear resistance and to repair damaged blades by filling voids and rejuvenating geometry. Hardfacing (fusion surfacing, weld surfacing) is one such technique that relies on a metallurgical bond between the coating and the substrate.[1-3] Either an overlay method, which includes plasma spraying and plasma transferred arc (PTA) processes, or a welding method, encompassing tungsten inert gas (TIG) and laser processes, is used to produce the hardfacing deposit.[4] While hardfacing may influence the microstructure of base (substrate) materials, it can provide better wear resistance, depending on the coating materials. Typically, a higher wear resistance can be found in harder materials, with the drawback of lower fracture toughness. Desirable properties for high-temperature wear resistance include high hot hardness, resistance to oxidation at elevated temperature, and a high thermal diffusivity.[5] For intense operating environments, Ni-based superalloys do not have significant wear or galling resistant properties. For this reason, Co-based alloys are used in coatings to provide wear resistance.[6] Co-based alloys can be tailored for wear applications by increasing the carbon content and adding more solid solution strengthening elements to better maintain the high temperature hardness.[7] Two commercial Co-based hardfacing alloys, Stellite 694 and Tribaloy T-800, are being considered for wear protection of IN738 turbine blades in this study. Wear resistance of the two alloys is derived from two very different microstructural features. Stellite 694 has a high carbon and tungsten content, relying on carbides and solid solution strengthening, while T-800 has a high molybdenum content leading to a high volume fraction of an intermetallic Laves phase.

In this study, IN738 is examined, as it is a typical polycrystalline blade alloy used in high-temperature applications. IN738 is strengthened primarily by γ'-Ni_3 (Al, Ti) and MC (M = Ti, Nb, Ta, V, Hf, or Zr) carbide precipitates in a γ matrix. MC carbides are transition metal carbides formed during solidification typically from Ti, Nb, Ta, and of course, C.[8] These carbides can greatly affect the mechanical properties of the material, frequently growing in interdendritic and grain boundary regions. Factors controlling the growth of these carbides are the following: microsegragation levels of the carbide forming elements, morphology of the γ matrix, and local solidification time. For the coarse dendritic growth characterized by the vacuum investment casting process used to manufacture IN738 turbine blades, a script type morphology MC carbide is produced.[9] The γ' phase volume fraction for polycrystalline IN738, in the aged condition, is usually between 40-45%[10] and provides the material with comparable temperature-

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Table 1 Chemical Composition (wt.%) of IN738

Cr	Co	Ti	Al	W	Ta	Mo	Nb	Zr	B	C	Ni
15.7-16.3	8.0-9.0	3.2-3.7	3.2-3.7	2.4-2.8	1.5-2.0	1.5-2.0	0.6-0.11	0.06-0.10	0.008-0.10	0.09-0.13	bal

Table 2 Chemical Compositions (wt.%) of the Hardfacing Alloys

Alloy	C	Si	Mn	Cr	Ni	W	V	Fe	B	Mo	Co
Stellite 694	0.85	1	1	28	5	19.5	1	3	0.1	0	bal
Tribaloy T-800	0.08	3.4	0	17.5	1.5	0	0	1.5	0	28.5	bal

Table 3 Sample Mixture Ratios

Sample	694, wt.%	IN738, wt.%	T-800, wt.%
1		100	
2	100		
3	5	95	
4	25	75	
5	50	50	
6	75	25	
7			100
8		95	5
9		75	25
10		50	50
11		25	75

dependent deformation mechanisms as single crystal superalloys with high (60-80%) γ' volume fractions.[10] Processes that involve local melting or heating followed by rapid solidification, such as grinding or welding, can dissolve the γ' precipitates leading to a layer that lacks this phase.[11] A post process heat treatment is required to partially restore the material's properties.

To understand the performance of the coatings, particularly the interface, this study is carried out to examine the phase and morphology changes resulting from interface mixing between a hardfacing deposit and the blade/substrate. Alloys with different mixture ratios of substrate and coating materials are investigated for existence of topologically close-packed (tcp) and deleterious carbide phases using optical microscopy, differential scanning calorimetry (DSC), scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS).

2. Experimental

2.1 Materials

IN738 was provided as a fine powder, the composition of which is given in Table 1. The Stellite 694 and Tribaloy T-800 were in the form of plasma transferred arc (PTA) hardfacing pre-alloyed powders. The compositions are given in Table 2. The representative interface mixture samples were created by mixing each hardfacing alloy with the IN738 substrate. The mixing ratio is detailed in Table 3. The powders were mixed mechanically in small quantities and specimens were cast in alumina crucibles during DSC analyses. Two heating cycles up to 1500 °C, under an inert argon atmosphere, were used to ensure complete mixing, creating "new" alloys. The DSC process is described in the next section.

2.2 Differential Scanning Calorimetry

Thermal analyses were performed using a Netzsch DSC 404C Pegasus (Netzsch-Geratebau GmbH, Selb, Germany). Alumina crucibles were used for reference and sample containers, the reference crucible was left empty. Lids were also used to prevent possible elemental loss during the process. Powder mixture samples were measured into 35-40 mg specimens. Masses of each specimen and both crucibles are recorded before and after each DSC run. With the specimen and reference crucibles in place, the furnace cavity is evacuated to high vacuum using a fore (mechanical) pump and a turbomolecular drag pump. Testing under vacuum can be done; however, an inert atmosphere was used to hinder the loss of more volatile elements, in addition to the use of the lid. Argon at 50 ml/min is used to backfill and maintains a slightly positive pressure within the furnace; it is also used as the furnace protective gas. Heating and cooling are controlled through a TASC 414/4 controller (Netzsch-Geratebau GmbH, Germany) linked to PC-based software, where the segments of the heating/cooling program can be set. The heating/cooling rate was set to 20 °K/min with a 10 min hold at the 1500 °C maximum and a 5 min hold at 400 °C between the two cycles. After the second cycle, the cooling rate is not controlled below 400 °C. Output to Netzsch data analysis software produces plots of DSC signal (mW/mg) versus temperature (°C). Of utmost concern for this study is the second cycle cooling curves, which provide an indication of the number of phases precipitated in the substrate/hardfacing alloy mixture based on the number of exothermic peaks.

2.3 Metallography and Compositional Analysis

Due to the small size of the DSC specimens, all the specimens for each material combination were mounted together into a single Bakelite sample. The samples were prepared using conventional metallographic procedures; Marble's reagent was used for etching (10 g $CuSO_4$, 50 ml HCl, 50 ml H_2O). Final preparation included ultrasonic cleaning and the application of a carbon surface film for conductivity when analyzing the microstructure using SEM. Phase and morphology characterization was done using an Olympus PME 6 optical microscope

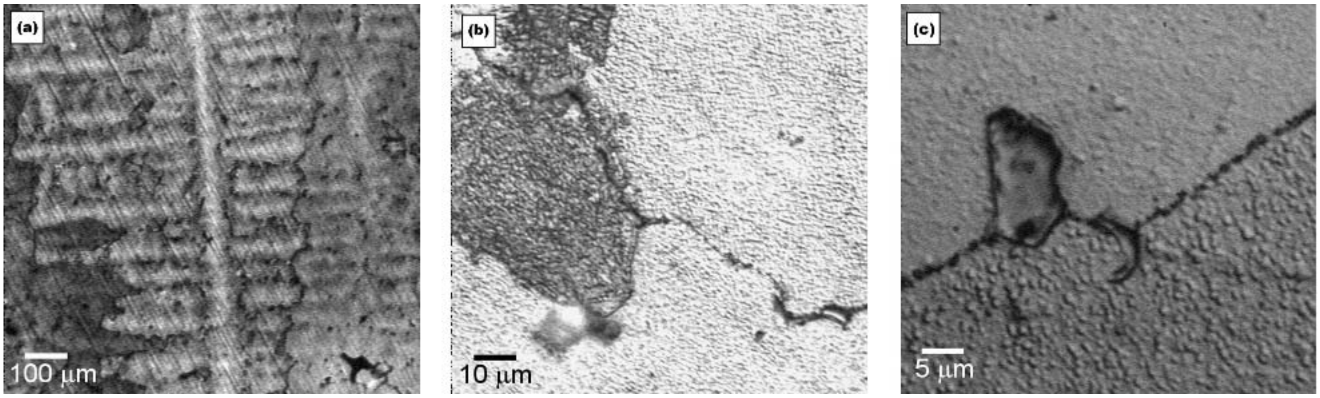

Fig. 1 IN738 blade sample microstructure: **(a)** dendrite in equiaxed grain structure, **(b)** grain boundary and precipitates, **(c)** grain boundary carbide and precipitates

(Olympus Optical Co. Ltd., Tokyo, Japan) and a JSM-6400 digital scanning electron microscope (JEOL USA Inc., Peabody, MA) in backscatter (BEI) and secondary electron (SEI) modes. Qualitative compositional analysis was performed on the same specimens using a LINK eXL energy dispersive x-ray LZ-4 microanalysis system (Oxford Analysis Instrumentation, Eynsham, Oxford, UK), at a voltage of 20 kV. Visual phase differentiation was performed on screen and with photomicrographs. Specific areas were then selected to acquire an EDS spectrum to verify compositional differences between visually identified phases. A final check was done by comparing the number of phases found using SEM/EDS techniques and the number indicated from the DSC analyses.

3. Results

3.1 IN738

Sections of an investment cast IN738 turbine blade were cut for a preliminary analysis of the microstructure. Several micrographs were taken from different locations on the blade and at varying magnification to understand the typical structure of the IN738 material. Figure 1 shows some common features that characterize IN738, i.e., large equiaxed dendritic grain structure, jagged grain boundaries, high γ' volume fraction, and grain boundary carbides. Differences were observed between the root and tip sections of the blade. The tip exhibits a much finer dendritic structure compared with the root of the blade. Also, casting defects were more prevalent at the tip of the blade; more and larger interdendritic voids and porosity were found toward the blade tip.

3.2 Stellite 694

Microstructure of the Stellite 694 hardfacing alloy is shown in Fig. 2(a), along with the DSC curve for this material in Fig. 2(b). Evident in Fig. 2(a) are three main constituents: matrix (α, Co base), primary carbides distinguished by white contrast (later confirmed to be WC), and eutectic α + carbides. The DSC curve indicates four peaks that occur at various temperatures in the cooling cycle; hence a fourth microstructural constituent may exist in Stellite 694. It can be speculated that another type of carbide is present and the carbide could be $M_{23}C_6$, M_7C_3, or MC type precipitating from α-Co in addition to the primary WC during cooling. Peaks in the heating (upper) curve show the allotropic transformation characteristic for cobalt as well as two melting peaks indicating the presence of two distinct phases when in powder form.

A detailed microstructural study was conducted on the specimen cast with 50 wt.% Stellite 694 + 50 wt.% IN738. A completely different microstructure was observed (Fig. 3a) compared with the 100% Stellite 694 specimen in Fig. 2(a). There is a larger percentage of matrix (Co + Ni solid solution) shown in Fig. 3(a) for the 50%/50% mixture, and the precipitates exhibited primarily a script-type morphology. A qualitative EDS analysis Fig. 3(c) and its distinct morphology suggests that the script type phase could be (W, Ti)C. In addition to the script phase, another group of phases with eutectic features were found at higher magnifications, as shown in Fig. 4(a). EDS analyses were conducted at three different locations with distinctive contrasts (indicated by 1, 2, and 3 in Fig. 4a) and varying degrees of compositional difference were found in the spectra [Fig. 4 (b-d), respectively]. Phases 1 and 3 are clearly Cr-rich phases that contain W, while phase 2 is a W-rich phase with some Cr. While there is some indication of the presence of C, it is not certain at this stage whether these phases are carbides or intermetallics. They do, however, differ significantly from the primary script type phase [(W, Ti)C]. Parallel with the SEM and EDS data, the DSC cooling curve, shown in Fig. 3(d), suggests there could be four phases in the Co-Ni matrix. The positive nature of these phases will be further examined in the discussion section.

To demonstrate the gradual change in the microstructure due to the variation in the mixing ratio, optical micrographs at both low and high magnification were assembled together, producing Fig. 5. The change in quantity, morphology, and nature of the phases can be observed in the precipitates from left to right in the figure. It also provided an exaggerated view of possible interface microstructures between the surface coating and the substrate.

3.3 Tribaloy T-800

Unlike the Stellite 694, the 100% Tribaloy T-800 alloy specimen shown in Fig. 6 reveals only matrix and Laves in-

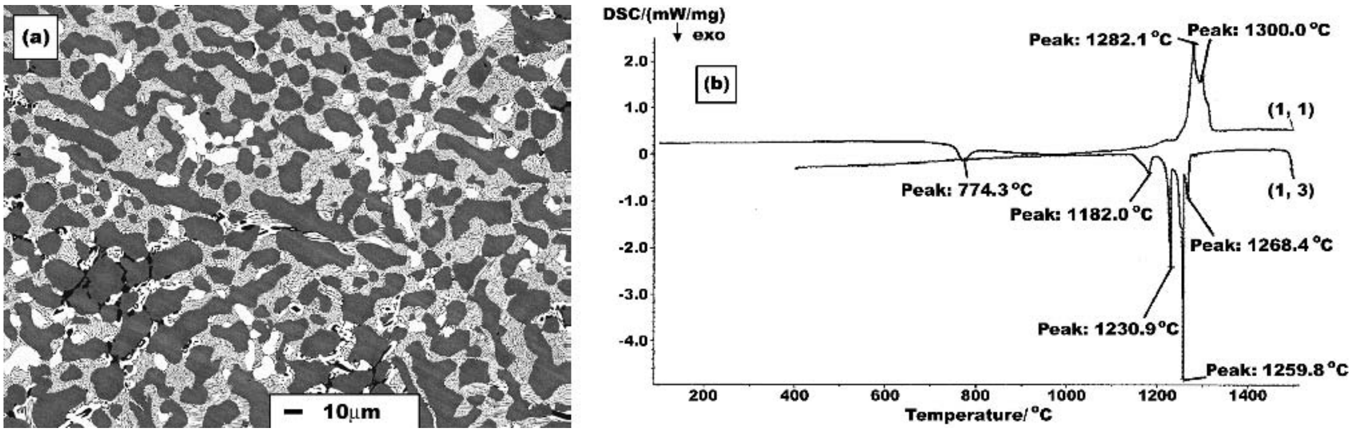

Fig. 2 **(a)** Stellite 694 hardfacing alloy at low magnification showing multiphased microstructure high in carbides, **(b)** DSC curve for Stellite 694

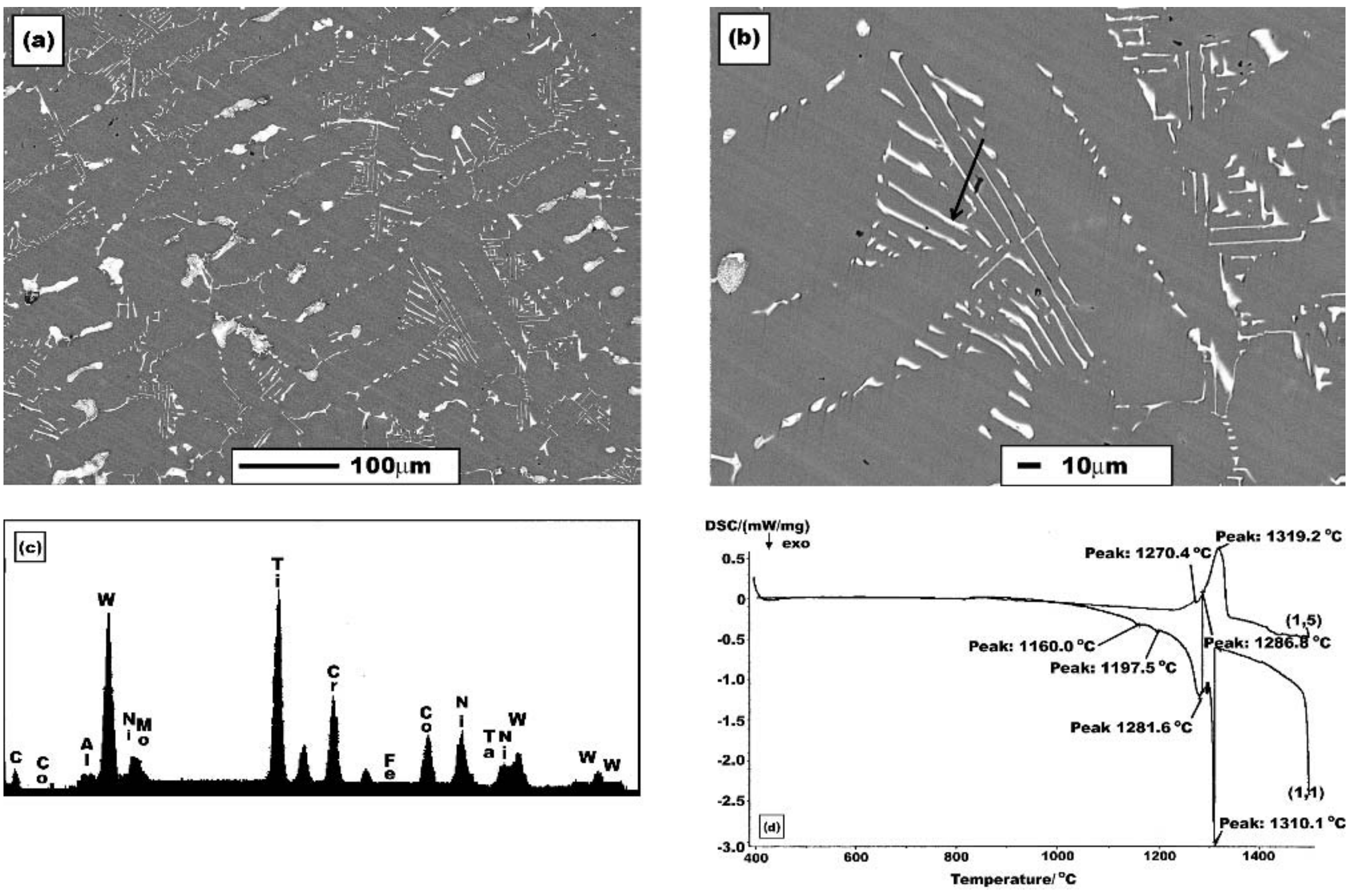

Fig. 3 **(a)** 50% IN738 with 50% Stellite 694 mixture at magnification 200; **(b)** 50% Stellite 694 at magnification 600, arrow indicates location of analysis; **(c)** EDS spectrum from location shown in 3b; **(d)** DSC curve for 50% IN738 + 50% Stellite 694

termetallic phases. The identification of the Laves phase is based on the composition of the alloy and the research by Wu and Redman.[12] With the addition of 50 wt.% IN738 into the T-800 alloy, the microstructure evolved from a blocky shaped Laves phase in Fig. 6 to a more eutectic form of the Laves phase (Fig. 7a) and the emergence of carbides, shown in Fig. 7d. EDS analyses show a typical Co-Ni-Cr solid solution matrix (point 1 in Fig. 7a detailed in Fig. 7b) and possible Laves phase $(Co, Si)_2Mo$ alloyed with Cr and Ni. The presence of carbides was revealed through examination at higher magnification (Fig. 7d and 7e). These observations of microstructural constituents correspond well with the DSC cooling curve in Fig. 7(f), where three distinct peaks are present. While the sequence of formation for these microstructural constituents is

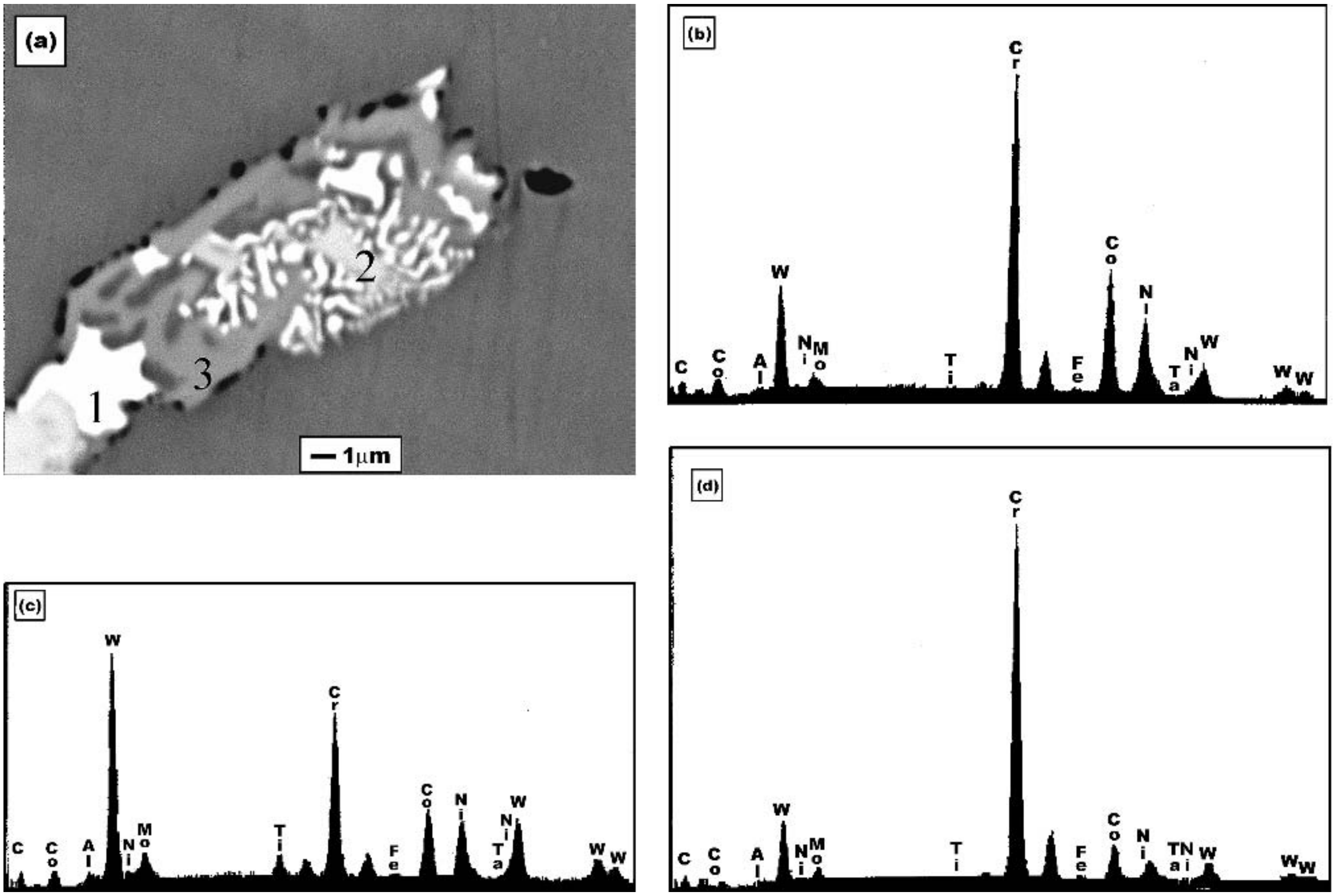

Fig. 4 **(a)** 50% Stellite 694 at magnification 4000 with numbers indicating locations of analysis, **(b)** EDS spectrum from location 1 in (a), **(c)** EDS spectrum from location 2 in (a); **(d)** EDS spectrum from location 3 in (a)

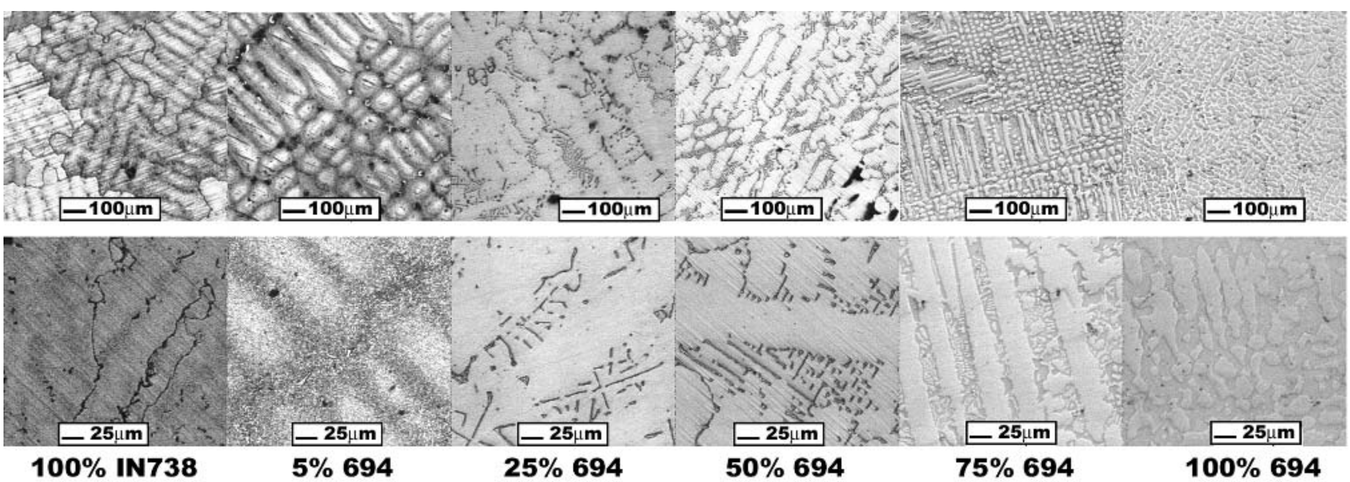

Fig. 5 Morphology evolution between IN738 and Stellite 694

not known, the three phases can be identified as carbides, Co-Ni-Cr matrix, and Laves phase.

A further increase in T-800 content up to 75 wt.% provided microstructures with a combination of Fig. 6 (100% T-800) and Fig. 7a (50% T-800), i.e., blocky Laves (points 2 and 3 in Fig. 8a and 6) and eutectic Laves (point 4 in Fig. 8a and 7a), in addition to a Co-Ni-Cr based matrix (point 1 in Fig. 8a). No carbides were observed with this composition (Fig. 8d); however, the presence of carbides cannot be ruled out. The DSC cooling curve clearly shows the formation of only two phases

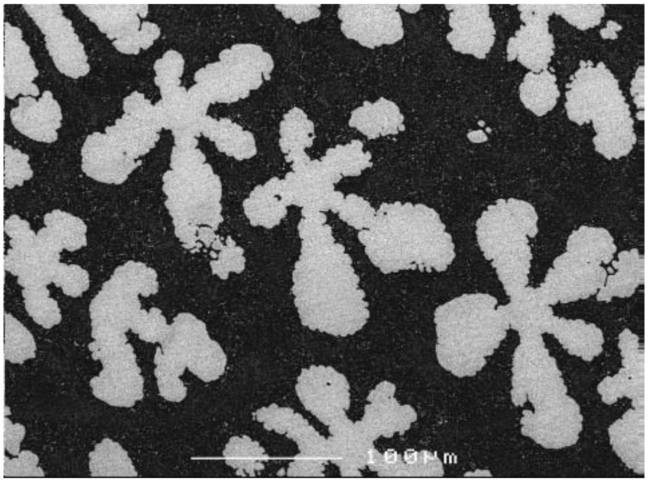

Fig. 6 Tribaloy T-800 hardfacing alloy at magnification 300 showing characteristic Laves phase

during cooling (Fig. 8e), which supports the microstructural and EDS observations.

4. Discussion

Co-based alloys were originally designed for castings in wear resistant applications and have subsequently been used as hardfacing alloys applied to components requiring added wear resistance. From a microstructural point of view, there are three types of Co-based hardfacing alloys.

The first type is a Co solid solution, which typically contains <0.4% carbon. The solid solution type Co alloys are rarely used for wear resistant coatings; rather, they are used for good corrosion and thermal fatigue resistance.

The second type of Co-based hardfacing alloys contains significant amounts of carbides with Cr, W, and C intentionally added into the matrix for increasing and retaining high-temperature hardness. The microstructure for Stellite 6 with a composition of Co-1.2C-29Cr-4.5W exhibits a hypoeutectic dendritic structure of $M_{23}C_6$ carbides (M = Cr, W, Co) and cobalt solid solution.[12] Other phases, such as Cr_7C_3 and Co_3W, have also been found in Stellite 6, for example, in addition to the primary $M_{23}C_6$ carbides.[13] The addition of Si in Stellite 6 has resulted in the formation of μ and σ phases,[14] a hybrid form of type 2 and type 3 Co-based wear-resistant alloys. Stellite 694, examined in this study, is one of the carbide containing Co alloys with a greater W content than Stellite 6. The refractory elements such as W and Mo, while known for their effective solid solution hardening effect, contribute to the formation of MC, M_6C, and intermetallic phases such as $Co_3(Mo,W)$.[15]

A third type of Co-based hardfacing alloy uses intermetallic phases, such as Laves phase $(Co, Si)_2Mo$, to impart wear resistance. The most common alloys in this category are those in the Tribaloy series; the T-800 alloy used in this study falls within this group. Coatings containing this Laves phase are unique in terms of its high temperature wear resistance, as the Laves phase retains its stability up to 1230 °C. Hence, these materials have found wide application in coating components for high temperature wear resistance. However, the alloy is quite brittle due to the presence of the Laves phase, and the weldability is quite poor. A considerable amount of cracking has been observed during both TIG and PTA hardfacing processing.[16] Process optimization is underway by the present authors to develop optimal PTA hardfacing parameters.

4.1 Stellite 694 + IN738

As described in the results section, three prominent phases were observed: the Co matrix, primary carbides, and eutectic carbides/Co in the cast Stellite 694. The nature of the carbides and their crystalline structure are being examined at present and will be reported in a future publication. An allotropic phase transformation at 774 °C, as shown in Fig. 2(b), suggests that the Co matrix exhibits a hexagonal close-packed (hcp) structure at room temperature for the pure Stellite 694 alloy. With the addition of IN738, the high-temperature face-centered cubic (fcc) structure is stabilized by Ni and Fe, and no allotropic phase transformation can be seen. Figure 3(d), for example, shows this for all the mixtures of Stellite 694 and IN738 studied.

Precipitate morphology was drastically modified when Stellite 694 was mixed with IN738. By comparing Fig. 2(a) and 3(a), it can be seen that the total amount of precipitates have been reduced due to the composition changes. Most of the precipitates exhibit a script-type shape with the occasional occurrence of eutectic islands (Fig. 4a).

It is believed from the EDS analysis that the script type precipitates are carbides containing large percentages of W, Ti, and Cr. It is not certain at present whether it is a $M_{23}C_6$, M_7C_3, or MC type carbide; therefore, x-ray diffraction (XRD) and wavelength dispersive spectroscopy (WDS) are being conducted to reveal the nature of the carbides.

While the occurrence of a script type precipitate, particularly a tcp phase, may be detrimental to the mechanical properties of the coated material,[8] the possibility of the formation of intermetallic phases is of great concern. With the elevated W content in Stellite 694 and the addition of Mo from the substrate alloy IN738, the mixture may be prone to the formation of intermetallics.

The stability of the matrix can be characterized by the electron vacancy theory embodied in the PHACOMP (an acronym for phase computation) numerical calculation algorithm.[8] PHACOMP has been used very successfully in the modification of chemical compositions for both Ni- and Co-based alloys to avoid the formation of tcp phases.

PHACOMP calculations based on the methods described in Ref. 8 and 17 concluded that the mean Nv numbers are 2.47 and 2.57 for 25% and 50% mixtures of Stellite 694 in IN738, respectively, indicating the susceptibility of forming tcp phases. Further analysis using SEM/EDS suggested the existence of tcp phases. As shown in Fig. 4(a), the island-shaped constituent in fact contains several microscopic features, i.e., the bright blocky phase (labeled 1), the greyish phase (labeled 3), and the eutectic-like phase (labeled 2). It is speculated that phase 2 (containing W) evolves from phase 3 and depletes it of W, as seen in Fig. 4(d). While both phases 1 and 3 contain

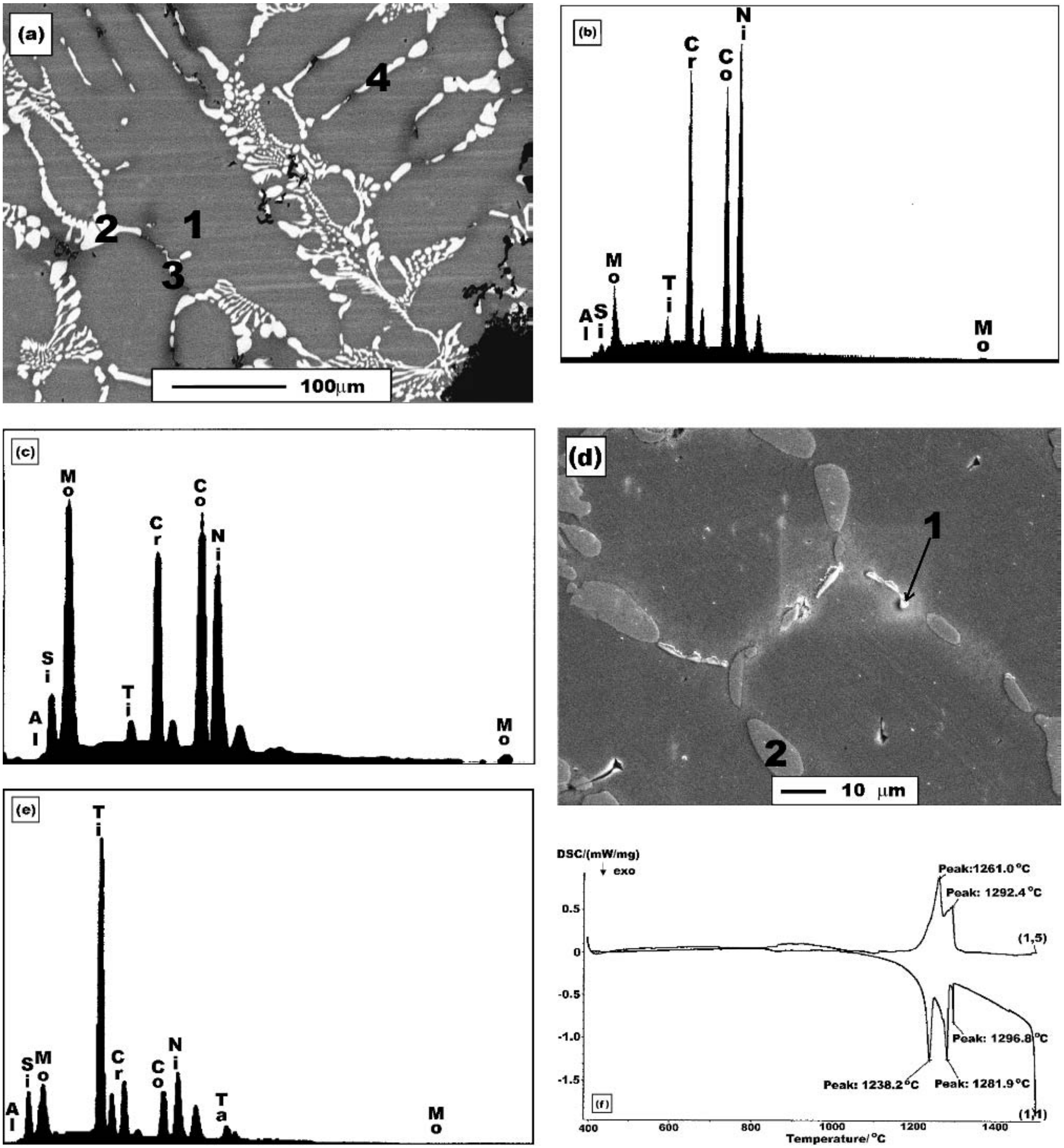

Fig. 7 **(a)** 50% IN738 with 50% Tribaloy T-800 mixture at magnification 300 with numbers showing locations of analysis, **(b)** EDS spectrum from location 1 in (a), **(c)** EDS spectrum from locations 2, 3, and 4 in (a), **(d)** 50% T-800 at magnification 1000 in SEI mode with arrow indicating location of analysis, **(e)** EDS spectrum from location shown in (d), **(f)** DSC curve for 50% IN738 + 50% Tribaloy T-800

similar chemical elements, phase 3 seems to be lower in W due the formation of phase 2. It is likely that both phase 1 and 3 are carbides in nature and phase 2, formed from phase 3, is a tcp phase as predicated by PHACOMP theory.

The possibility of forming a tcp phase in the interface between the hardfacing deposit and substrate is of concern when developing a hardfacing process technique. The formation of a tcp phase in the interface could potentially cause a loss of strength and ductility at service temperature, as well as loss of low temperature ductility.[8] A judicious decision shall be made

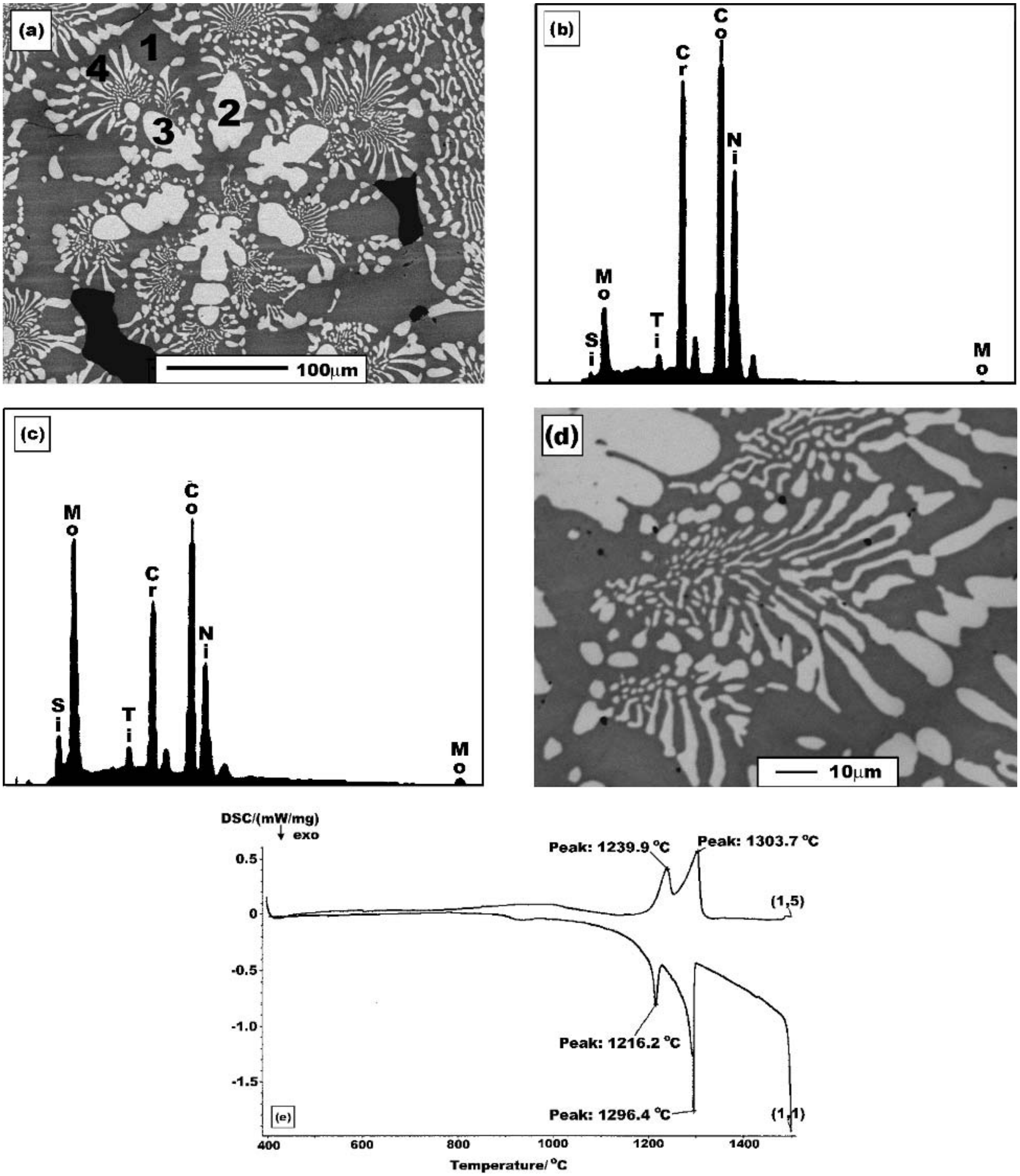

Fig. 8 **(a)** 25% IN738 with 75% Tribaloy T-800 mixture at magnification 300 with numbers showing locations of analysis, **(b)** EDS spectrum from location 1 in (a), **(c)** EDS spectrum from locations 2,3,and 4 in 8a, **(d)** 75% T-800 at magnification 1000, **(e)** DSC curve for 25% IN738 + 75% Tribaloy T-800

when choosing the process methods (TIG, PTA, or laser) and process parameters to avoid or minimize the formation of tcp phases.

A preliminary study conducted by the authors[16] showed that the PTA hardfacing process is capable of producing a reduced intermixing ratio (or dilution) when compared with the TIG process. Therefore, the potential of forming tcp phases can be minimized.

4.2 Tribaloy T-800 + IN738

Tribaloy T-800, a Laves type Co alloy, is less susceptible to the detrimental effect from the formation of tcp phases due to mixing with substrate superalloys. Consideration should have been taken when selecting this hardfacing alloy to allow for reduced ductility. As seen in Fig. 6, a typical Laves phase in Co matrix is formed during cooling and is well distributed throughout. During deposition of T-800 onto the substrate, intermixing (or dilution) will occur. Reported in one study on laser cladding using T-400 (similar composition to T-800, however with lesser Cr and Si contents) onto a steel substrate, as high as 12% coating material dilution was observed,[15] while 4% dilution on IN 718 substrate was observed. Due to the dilution of the hardfacing material, the microstructure of the diluted area contains Co_3Mo_2Si Laves phase in the case of steel substrate. CoMoSi Laves phase was found when the substrate was IN 718.

In this study, the mixture with 75% T-800 and 25% IN738 produced microstructure containing primarily two phases: the Co matrix and the Laves phase (Fig. 8a and 8d). The Laves phase is confirmed based on both previous microstructural observation of Tribaloy and EDS spectrum showing a prominent Si peak in Fig. 8(c).

With an increase in the IN738 content to 50%, the blocky shaped Laves phase subsides. Instead, both script and eutectic forms of the Laves phase are present, as shown in Fig. 7(a). All the grain boundaries are heavily populated with precipitates as seen in Fig. 7(d), and the morphology of phase 1 in Fig.7(d) resembles carbides. EDS analyses of both phases 1 and 2 were carried out with phase 1 containing mainly Ti while phase 2 showed a similar spectrum as Fig. 7(b), i.e., Laves phase. The precise chemical composition of all phases will be analyzed using SEM/WDS and detailed results will be reported subsequently.

5. Conclusions

Based on the results presented, the following conclusions can be drawn for this study:

(a) Deleterious phases and generally undesirable microstructures are formed in alloys with mixture ratios of 25 wt.% and 50 wt.% of the hardfacing component with IN738.
(b) Stellite 694 develops a more severe script type phase in the 50%/50% mixture compared with Tribaloy T-800 with a eutectic and blocky type microstructure.
(c) Results from DSC cooling curves correlated well with SEM/EDS data. Knowing the number of peaks on the cooling curve for specific mixtures aided in the search for multiple phases using the SEM.
(d) A hardfacing process that minimizes mixing between the deposit and substrate is more desirable.

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