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COMPARISON OF THE OXIDATION FEATURES OF E110 ALLOY AND SINTERED SILICON CARBIDE ON E110 ALLOY AT 1200 ^{0}C IN AIR FOR FUEL CLADDING STUDIES

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СРАВНЕНИЕ ОСОБЕННОСТЕЙ ОКИСЛЕНИЯ СПЛАВА Э110 И СПЛАВА Э110 ^{0}C ПОКРЫТИЕМ ИЗ КАРБИДА КРЕМНИЯ ПРИ 1200 ^{0}C НА ВОЗДУХЕ ДЛЯ ИСПОЛЬЗОВАНИЯ В ОБОЛОЧКАХ ТЕЛОВЫДЕЛЯЩИХ ЭЛЕМЕНТОВ

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Аннотация. *Защитные покрытия предназначены для снижения окисления при экстремальных температурных условиях реактора. Данное исследование включает нанесение покрытия SiC на подложке Э110 (Zr-1Nb) в атмосфере воздуха и аргона методом селективного лазерного спекания (SLS). Испытание на высокотемпературное окисление проводили на образцах при температуре 1200 ^{0}C на воздухе в течение 600 секунд. Масса, полученная после испытания на высокотемпературное окисление, была рассчитана как 34,6 мг/см2, 31,1 мг/см2 и 22,8 мг/см2 для непокрытого образца сплава, Э110 с покрытием SiC- Э110, спеченного на воздухе, и с покрытием SiC- Э110, спеченного в атмосфере аргона соответственно. Рентгеноструктурные фазовые исследования показывают образование более 90% ZrO2 на сплаве Э110 без покрытия. SiO2 и YAlO3 составляли более 50% оксидов, образующихся на поверхности образцов, покрытых спеканием порошка SiC, после испытания на высокотемтературное окисление. Таким образом, покрытие SiC на циркониевом сплаве может играть значительную роль в снижении окисления, особенно в покрытиях, наносимых в инертной среде. Полученные результаты можно рассматривать как задел для дальнейших исследований преставляемых покрытий для защиты оболочек ядерного топлива.*

Introduction. Selective Laser Sintering (SLS) is an Additive manufacturing (AM) 3D printing technology which is rapidly gaining applications in various fields [1]. This application comprises ceramics for fuels as well as structural components of nuclear reactors [2]. A typical advantage of selective laser sintering includes rapid prototyping and manufacturing objects of complex geometries. *SiC* materials, such as monoliths and composites, are promising materials for nuclear fuel cladding for LWRs and advanced reactors, components of fuel particles and pellets for reactor core structural components in fission reactors, including functional structures for fusion reactors [3]. Those applications depend on the central properties of *SiC*, such as excellent strength at higher temperatures, chemical inertness, comparatively low neutron absorption, and its stability to

neutron irradiation up to high doses. The ability of *SiC* resistance to high-temperature as well as high-radiation environment proves *SiC* a candidate material for cladding fuels in light water reactors [4]. *SiC* remains intact even at temperatures beyond 1500 ^{0}C [5] and also parasitically captures fewer neutrons when compared to zircaloy as well as possessing very low activation [6].

Protective coating on zirconium alloys has been a second choice proposed approach to enhancing Accident Tolerant Fuel (ATF) cladding concept [7]. The aim of this study is to investigate sintered *SiC* on *Zr-1Nb* alloy substrate under high temperature oxidation in air atmosphere in order to compare the oxidation behavior of sintering performed in air and argon SLS chamber.

Research methods. *Zr-1Nb* (E110) substrates of dimensions (20mm x 20mm x 2mm) were polished and wiped with an acetone filled cloth. An SLS system (IPG Photonics, Moscow, Russia) equipped with *Yb* fiber of output wavelength and maximum laser power of 1070 nm and 500 W respectively was used. This was then followed by sintering *SiC* (SIKA DENSITEC L) micro composite with minor phases including *Al2O3* and *Y2O3* onto the *Zr-1Nb* alloy. The Fig. 1 and Table 1 represent the SLS system used and parameters followed in the deposition process respectively.

Table 1

SLS coating parameters

Samples	Laser power (W)	Scanning time (µs)	Laser speed (mm/s)	Coating thickness (µm)	SLS Chamber Atmosphere
b, c / d, e	125	500	25	200	Air/Ar

The sample 1 and sample 2 involve sintering of SiC on both sides of the substrates under air and *Ar* atmosphere respectively. After the sintering, each sample was cut into two parts and each from a sample was drilled through for flexible fixing into the furnace. The furnace was heated to the temperature of 1200 ^{0}C which was accompanied by placing the samples into the heated furnace for 600 s and quenched after that in air. X-Ray Diffraction (XRD) phase composition was investigated with XRD 7000 diffractometer maxima (Shimadzu, Kyoto, Japan) along with the Sleve+ program. Qualitative and quantitative analyses were done with the Crystallographica Search-Match and PowderCell24 together with PDF-4+ database respectively.

Results. Table 2 and Fig. 1 represent the results obtained after High Temperature Oxidation (HT-Ox) tests and the macrographs of the individual samples before and after the HT-Ox tests.

Table 2

Results of samples on mass-gain after HT-Ox test results

Samples	Description	Sintering atmosphere	HT-Ox	Mass distribution				
				Before (g)	After HT-Ox (g)	Δm (g)	Mass gain (mg/cm2)	PΔm, %
a	E110 alloy	-	oxidized	2,5110	2,6910	0,1800	34,62	7,17
b	SiC - E110 substrate	Air	oxidized	2,4719	2,6416	0,1697	31,19	6,87
c		Air						
d		Argon	oxidized	2,4894	2,6132	0,1238	22,75	4,97
e		Argon	-	-	-	-	-	-

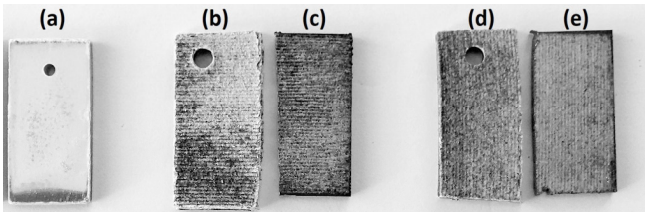

Fig. 1. Macrograph of samples; (a) Zr-1Nb substrate, (b) SiC – Zr-1Nb [air] and (d) SiC – Zr-1Nb [argon] oxidized at 1200 ^{0}C for 600 s, Un-oxidized samples (c & e)

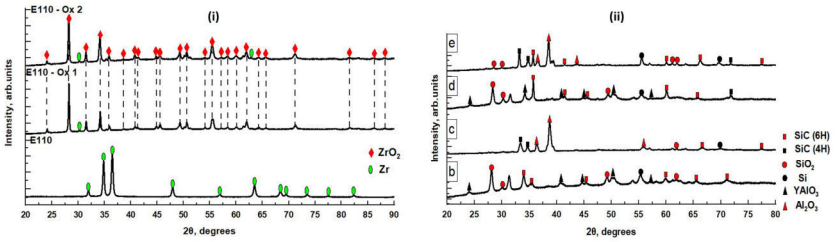

Fig. 2. XRD phase composition of samples: (i) Oxidized E110-alloy (sample a) (ii) Sample b, c, d, & e as described and indicated in Table 2 and Fig 1 respectively

Conclusion. *SiC* micro-composites were deposited on E110 alloy substrates and investigated under a high temperature oxidation environment in the air at 1200 ^{0}C for fuel cladding material studies. Experimental measurement of mass gain and XRD phase content analysis were conducted to study the behavior of the coating performed under air and argon atmosphere. The calculated mass gained and percentage mass change (*PΔm)* after the HT-Ox test was calculated to be 34,62 mg/cm2 (7,17%), 31,19 mg/cm2 (6,87%) and 22,75 mg/cm2 (4,97%) for bare *E110* alloy, *SiC-E110* sintered in air and argon SLS chamber respectively. XRD phase qualitative analysis reveals the formation of *ZrO2* above 90 % of the surface to sub-surface of the bare *E110* alloy. Also, *SiO2*, *YAlO3* became the main oxides yielding above 50 % and the remaining being *SiC* phases on the surface to sub-surface after HT-Ox test. The low outcomes of mass gained coupled with little changes in the dimensions of the *SiC* sintered sample fabricated in the *SLS* inert environment after HT-Ox proves optimistic and hence recommended for further research.

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