Interaction of $M_{n+1}AX_n$ Phases with Oxygen-Poor, Static and Fast-Flowing Liquid Lead-Bismuth Eutectic

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Abstract

Select MAX phase-based ceramics were screened with respect to their potential susceptibility to environmentally-assisted degradation in an oxygen-poor liquid lead-bismuth eutectic (LBE) environment, both under static and fast-flowing exposure conditions. The majority of the MAX phases exposed to oxygen-poor ($C_O \leq 2.2 \times 10^{-10}$ mass%) static liquid LBE for at least 1000 h at 500°C showed exceptional chemical compatibility with the heavy liquid metal, i.e., no evidence of LBE dissolution attack was observed, despite the absence of a continuous oxide scale on the surface of the exposed MAX phase ceramics. The local LBE interaction observed only with the Zr-rich MAX phases consisted in the partial substitution of Al by Pb/Bi in the MAX phase crystal structure and the in-situ formation of Pb/Bi-containing solid solutions. Moreover, the interaction of Zr-based MAX phases with static liquid LBE was accompanied by the dissolution of parasitic intermetallic phases, which facilitated the further LBE ingress into the ceramic bulk. The erosion resistance of select MAX phase ceramics was also assessed in oxygen-poor ($C_O \approx 5 \times 10^{-9}$ mass%) fast-flowing ($v \approx 8$ m/s) liquid LBE for 1000 h at 500°C. Despite the moderate LBE oxygen concentration, oxidation was the predominant corrosion mechanism, while no erosion damages were observed in the exposed MAX phase ceramics. The resistance of the MAX phase ceramics to both dissolution corrosion and erosion in contact with oxygen-poor

static and fast-flowing liquid LBE, respectively, was compared to that of the 316L reference structural stainless steel.

Keywords: MAX phase; Lead-bismuth eutectic (LBE); Liquid metal corrosion; Erosion

Highlights

- Exposing 11 MAX phases to oxygen-poor, static LBE showed little or no interaction.
- The LBE/ Zr_2AlC interaction formed a new $\text{Zr}_2(\text{Al,Pb,Bi})\text{C}$ MAX phase solid solution.
- The LBE/ $(\text{Zr,Ti})_{n+1}\text{AlC}_n$ interaction formed $(\text{Zr,Ti})_{n+1}(\text{Al,Pb,Bi})\text{C}_n$ solid solutions.
- $(\text{Nb,Zr})_4\text{AlC}_3$ showed the best erosion resistance in oxygen-poor, fast-flowing LBE.
- MAX phases outperformed 316L stainless steels exposed to similar LBE conditions.

1. Introduction

One of the six Generation IV (Gen-IV) nuclear reactor concepts currently under development is based on a lead-cooled design [1], which is commonly referred to as Lead-Fast Reactor (LFR), since fast neutrons (>1 MeV) are used to sustain the fission reaction. This high-energy neutron spectrum ensures a higher fuel efficiency, enabling the usage of depleted uranium as nuclear fuel and the transmutation of long-lived nuclear waste [2]. A prerequisite for the deployment of Gen-IV LFRs is the availability of qualified nuclear materials that can withstand the anticipated harsh service environment [3]. One of the important concerns vis-à-vis the development of Gen-IV LFRs is the compatibility of both structural and fuel cladding materials with the primary Pb-alloy coolant, such as liquid lead (Pb) or lead-bismuth eutectic (LBE). A good compatibility guarantees that all reactor components maintain their structural integrity and the materials used for component construction retain their properties in contact with this heavy liquid metal (HLM). Under certain operating conditions, mainly determined by the temperature, time and HLM oxygen concentration, C_O , these requirements are not met by most nuclear steel grades, which may either suffer from liquid metal embrittlement (LME), as in the case of ferritic/martensitic steels [4], or from HLM dissolution attack, as in the case of austenitic stainless steels. HLM dissolution attack involves dissolution of highly-soluble steel alloying elements, such as Ni, Cr and Mn, LBE penetration into the steel bulk, and ferritisation of the dissolution-affected zone due to the removal of the austenite stabilizers Ni and Mn [5,6]. One strategy to minimize liquid metal degradation phenomena relies on the use of corrosion inhibitors. These chemical substances are added in small amounts to the HLM and effectively decrease material loss rates by forming a protective film on the surface of both structural and fuel cladding steels [7]. Effective corrosion inhibitors that can be added to liquid LBE include oxygen, which forms a passivating oxide layer [8], or Ti or Zr, which have a high affinity for the N and C in the steel, thus forming a protective nitride or carbide layer on the steel surface [9]. However, maintaining a homogeneous concentration of corrosion inhibitors in large reactor systems is challenging and local depletion might occur.

An alternative corrosion mitigation approach aims at the development of innovative materials that are compatible with the HLM coolant over the entire range of nominal operation conditions and can withstand, to a certain extent, transient/accidental conditions. A group of ternary carbides and nitrides, known as the ' $M_{n+1}AX_n$ (MAX) phases', is considered to be intrinsically stable in contact with lead-based alloys. In $M_{n+1}AX_n$ compounds, M corresponds to an early transition metal, A is an A-group element (commonly group 13 or 14), X is C or N, and n is an integer equal to 1, 2 or 3 [10]. These nanolaminated materials combine properties typically associated either with ceramics and metals, i.e., they are elastically stiff and behave well at elevated temperatures ($>1000^\circ\text{C}$); they are good thermal and electrical conductors that are concomitantly damage-tolerant and machineable using conventional tools; and they exhibit a remarkable radiation tolerance above 600°C [10-13]. This combination of properties makes them attractive candidate materials for specific applications in HLM-cooled nuclear systems. The commercially available MAX phase-based materials Maxthal[®] 312 and Maxthal[®] 211 are mainly composed of Ti_3SiC_2 and Ti_2AlC , respectively, but do contain some parasitic phases, such as TiC and TiAl_3 . The corrosion behaviour of these materials has been previously investigated in contact with liquid Pb and LBE [14-18]. An overview of the test conditions and the reported interaction is provided in Table 1. The four main test parameters are temperature, exposure time, HLM C_O , and HLM flow velocity. Overall, the reaction was very limited and consisted mainly of surface oxidation, resulting in the formation of a few micrometres-thick TiO_2 (rutile) scale. The most severe degradation was observed in Ti_2AlC after an exposure of 10,000 h at 650°C to static LBE with $C_O \approx 10^{-8}$ mass%, where LBE was detected between the MAX phase grains, indicating that the intermetallic phase present in the commercial Maxthal[®] 211 grade partially dissolved [15]. This underlines the importance of minimizing the amount of parasitic phases in these materials. The MAX phases are considered as candidate structural materials for pump impeller components in Pb- and LBE-cooled reactors, such as the irradiation facility MYRRHA (multi-purpose hybrid research reactor for high-tech applications) currently under development at SCK•CEN, Belgium [19]. Pump impellers should operate reliably at system-specific temperatures (e.g., $\sim 480^\circ\text{C}$ [18] and $\sim 270^\circ\text{C}$ [20] for Pb- and LBE-cooled reactor systems, respectively) in contact with a fast-flowing HLM. This implies that they should withstand liquid metal corrosion and, most importantly, erosion, as the HLM flow velocity is expected to reach 10-20 m/s locally on the impeller surface [20]. Despite its importance for the envisaged pump impeller application, no dedicated study of the effect of fast-flowing LBE on the performance of MAX phase-based materials has so far been published.

Recently, Bentzel et al. studied the behaviour of several MAX phases in static liquid sodium, another Gen-IV coolant, up to 750°C for 168 h [21]. They reported that Ti_3SiC_2 , Ti_2AlC and Cr_2AlC were stable, as well as their grain boundary phases. Ti_3AlC_2 , however, showed signs of degradation along the grain boundaries and Al-depletion in the MAX phase grains [21]. Additionally, it should be mentioned that one of the methods to synthesize Pb-containing MAX phases in the 1960s – Pb is a candidate A-element – consisted of exposing a powder mixture of Ti and TiC to liquid Pb at 850°C for 600 h [22]. This fact indicates that certain MAX

phases might be even more stable in these metallic coolants than their corresponding binary carbides, e.g., TiC.

The primary goal of this study is to identify MAX phase compounds that do not suffer liquid metal degradation in oxygen-poor, fast-flowing liquid LBE, hereby targeting the harsh service conditions of pump impeller components. Therefore, the intrinsic stability of 11 different MAX phase-based materials was assessed first in oxygen-poor, static LBE. The selected exposure temperature was 500°C, so as to be conservative for both liquid Pb and LBE coolants, while the tests lasted for at least 1000 h, which is a standard exposure time for initial material screening. The set of tested materials comprised the commercially available Maxthal[®] 312 and Maxthal[®] 211 grades, as well as lab grades of MAX phase ternary carbides and solid solutions thereof. Ti, Zr and Nb were selected as M-elements, as these elements have small fast neutron cross-sections [23]. Moreover, this work reports, for the first time, on the performance of MAX phases in contact with poorly oxygenated, fast-flowing liquid LBE at 500°C for 1000 h. The latter exposure condition is particularly suited to assess the erosion/corrosion behaviour of the tested MAX phase materials. The performance of the MAX phase-based ceramics is invariably compared with that of a reference structural steel, i.e., the 316L stainless steel, under similar exposure conditions.

Table 1. Overview of all MAX phase exposures to liquid LBE and Pb reported in literature.

MAX phase	HLM	T (°C)	Time (h)	C _o (mass%)	Flow Condition	Reported Interaction	Ref.
Ti₃SiC₂	LBE	700	1000	5 × 10 ⁻⁶	Static	No interaction observed	[14]
	LBE	550/650	3000	10 ⁻⁶ /10 ⁻⁸ (*)	Static	TiO ₂ (rutile) formation	[16]
	LBE	550/650/700	1000/3000/ 5000/10,000	10 ⁻⁶ /10 ⁻⁸	Static	TiO ₂ (rutile) formation – Local surface disintegration	[15]
	Pb	650/800	1000	10 ⁻¹²	Natural convection	No interaction observed	[17]
	Pb	550/600/750	500/2000/4000	10 ⁻⁶ /10 ⁻⁸ (*)	Static	TiO ₂ (rutile) formation	[16]
	Pb	500	2000	10 ⁻⁶	1 m/s	Indication of thin oxide scale	[18]
Ti₂AlC	LBE	550/650/700	1000/3000/ 5000/10,000	10 ⁻⁶ /10 ⁻⁸	Static	TiO ₂ (rutile) & Al ₂ O ₃ formation at C _o ≈ 10 ⁻⁶ mass% – Dissolution of parasitic phases at C _o ≈ 10 ⁻⁸ mass% and T = 650-700°C	[15]
	Pb	650/800	1000	10 ⁻¹²	Natural convection	No interaction observed with a freshly machined surface	[17]

(*) HLM oxygen content calculated as in [24]

2. Experimental Procedure

2.1. Exposures to Static LBE

Commercial Maxthal® 312 (nominally, Ti_3SiC_2) and Maxthal® 211 (nominally, Ti_2AlC) provided by Kanthal AB, Sweden, and a set of lab-made MAX phase grades were exposed to static liquid LBE at 500°C. Table 2 provides the list of MAX phase ceramics exposed to static LBE. For most of these materials, the synthesis procedure and as-produced material characterization has already been reported in literature. Additionally, Ti_3AlC_2 , $(\text{Ti,Nb})_2\text{AlC}$, Nb_2AlC and Nb_4AlC_3 discs were produced using a similar powder-metallurgical synthesis route similar to that reported in [25]; for this purpose, Ti (Chemetall, Grade S, <8 μm), Nb (HC Starck, <45 μm), Al (AEE, <5 μm) and C (Asbury, <5 μm) starting powders were mixed in near-stoichiometric ratios. The raw powder mixtures were first pre-compacted in a graphite die at 30 MPa and then spark plasma sintered at 1400, 1500, 1600 and 1650°C, respectively, under a load of 60 MPa for 5 min.

The MAX phase exposures to static LBE were carried out at SCK•CEN, employing a test setup presented in Fig. 1a and previously described by Lambrinou et al. [6]. The setup consisted of a stainless steel autoclave with an inner alumina (Al_2O_3) liner preventing contamination of the liquid LBE bath due to the dissolution of steel alloying elements from the autoclave walls. Temperature and LBE oxygen concentration were monitored during all exposures by a thermocouple type K and an electrochemical oxygen sensor ($\text{Bi/Bi}_2\text{O}_3$ reference electrode [26]), respectively. The LBE oxygen concentration was calculated as follows [6]:

$$\Delta U - \Delta U_{th} = -0.34756 + 2.5217 \times 10^{-4} \cdot T - 4.3087 \times 10^{-5} \cdot T \cdot \ln C_O \quad (1)$$

$$\Delta U_{th} = -6.91 \times 10^{-3} + 1.762 \times 10^{-5} \cdot T \quad (2)$$

where ΔU is the electric potential difference measured by the electrochemical oxygen sensor in V, ΔU_{th} is the thermoelectric potential between stainless steel tube and Mo lead wire in V, T is the absolute temperature in K, and C_O is the LBE oxygen concentration in mass%.

The aim of the performed exposures to static LBE was to assess the inherent resistance of the MAX phase ceramics to LBE dissolution attack. Therefore, the selected test conditions involved material exposure to oxygen-poor, static LBE ($C_O < 10^{-8}$ mass%) at a high temperature (500°C), so as to suppress the formation of protective oxide scales on the surface of the MAX phase samples, thus facilitating dissolution corrosion. The concentration of dissolved oxygen in the liquid LBE bath (~5 kg of freshly molten LBE) was maintained low throughout all exposures by continuously feeding a reducing gas (HYTEC 5: Ar-5% H_2 , Rapid Industrial Gases Ltd., UK) (Fig. 1a). These test conditions favour the occurrence of LBE dissolution attack in various nuclear grade structural steels, such as 316L stainless steels or similar [6,27]. One side of each MAX phase disc was mirror polished and studied by scanning electron microscopy (SEM) prior to the LBE exposure. Pieces of these discs were mounted on graphite plates using Mo wires (Fig. 1b); the graphite plates were used to maintain the MAX phases submerged in the LBE bath throughout the tests. Pictures of one graphite plate loaded with MAX phase samples are shown in Fig. 1b and Fig. 1c before and after the 3500-h exposure, respectively. In total, three different

screening exposures were performed, i.e., one for 3500 h and two for 1000 h. The exposure conditions (T , time and C_O) are summarised in Table 2. Fig. 2 shows the evolution of the LBE oxygen concentration as function of the exposure time. The 3500-h exposure shows a discontinuity around 500 h, which can be associated with lowering the bath temperature to remove a sample sub-set for analysis. Since the MAX phase samples were removed at $\sim 200^\circ\text{C}$, the oxygen uptake from the liquid LBE during the brief setup opening was minimal, with the LBE oxygen concentration quickly stabilising at $C_O \approx 10^{-10}$ mass% under the continuous purging with Ar-5% H_2 for the remainder of this exposure.

2.2. Exposure to Fast-Flowing LBE

The erosion resistance of select MAX phase ceramics was assessed in the CORELLA (Corrosion Erosion Test Facility for Liquid Lead Alloy) facility at KIT, Germany, in contact with fast-flowing LBE. A detailed description of this setup is provided in [30]. The CORELLA facility consists of a fast-rotating inner cylinder (rotation speed ≤ 2000 rpm) inside a container (test chamber) filled with the molten Pb-alloy, i.e., fresh, uncontaminated LBE in the present study. The flow velocity experienced by the samples depends on the angular velocity and radial distance from the centre, and the achievable LBE flow velocities can exceed 10 m/s. Detailed computational fluid dynamics (CFD) calculations on the flow patterns and HLM velocity may be found in [30]. Prior to actual testing, the liquid LBE was stored in another container (conditioning chamber), where it was conditioned in terms of C_O . Table 2 provides an overview of the tested MAX phase ceramics together with the exact test conditions. During testing, both T and C_O were monitored with thermocouple type K and electrochemical oxygen sensor ($\text{Bi/Bi}_2\text{O}_3$ reference electrode), respectively. The LBE C_O profile, for the exposure at the CORELLA facility, is also given in Fig. 2 as function of time. Due to a problem with data acquisition, the LBE C_O data from the first 300 h are missing. The tested MAX phase ceramics have been exposed to oxygen-containing ($C_O \approx 5 \times 10^{-9}$ mass%), fast-flowing ($v \approx 8$ m/s) liquid LBE at 500°C .

The MAX phase specimens had a thickness of 1.5 mm, a width of 15 mm, and a length of 45–60 mm. The front surface, facing the highest LBE flow velocity, was ground using a diamond grinding wheel (D46 SW-50-X2, TechnoDiamond). One sample of $(\text{Nb,Zr})_4\text{AlC}_3$ was machined by conventional milling to demonstrate the material's machinability and to assess the effect of higher surface roughness on erosion resistance. The MAX phase ceramics were tested together with the MYRRHA reference structural steel, i.e., the 316L austenitic stainless steel grade. For this purpose, a 316L steel specimen, made of the solution-annealed EUROTRANS-DEMETRA industrial-size plate (15 mm in thickness [6]; designated 316L SA in Table 2), was added to the set of specimens tested in the CORELLA. Before and after the exposure to fast-flowing LBE, the surface condition of all specimens was examined by profilometry (Talysurf-120L, Taylor Hobson). After the exposure, the solidified LBE residues were removed from the samples. The largest amount of LBE was removed in the liquid state by briefly immersing the samples in hot oil (Biskin sunflower and colza oil, 190°C , ~ 30 sec). Subsequently, the remaining LBE was removed in an equivolumetric solution of ethanol ($\text{C}_2\text{H}_5\text{OH}$), hydrogen peroxide (H_2O_2) and acetic acid (CH_3COOH).

2.3. Post-Exposure Characterization

All materials listed in Table 2 were cross-sectioned and their microstructure was studied in detail prior to and after exposure, using SEM (JSM-6610LV, JEOL, Japan; XL30-FEG & Nova 450 NanoSEM, FEI, The Netherlands) equipped with energy dispersive X-ray spectroscopy (EDS; Bruker and EDAX). Quantitative elemental analysis of select areas of interest was performed by electron probe microanalysis (EPMA; JXA-8530F, JEOL Ltd., Japan; operating conditions: 15 kV accelerating voltage, 15 nA beam current) using wavelength-dispersive X-ray spectroscopy (WDS). The reported chemical composition values are an average of 5 points, and the standard deviation is in the order of 1 wt%. Backscattered electron diffraction (EBSD; Hikari XP EBSD camera, EDAX, USA, on a FEI Nova NanoSEM 450, FEI, USA) was also employed to ensure that the MAX phase crystal structure was maintained in LBE-affected zones. A 200 kV transmission electron microscope (TEM; JEOL ARM200F, Japan) equipped with EDS and high-angle annular dark field (HAADF) detector was used in the scanning transmission electron microscopy (STEM) mode for elemental mapping and selected area diffraction (SAED) studies. TEM thin foils were prepared from specific areas of interest in the LBE-affected MAX phase ceramics by means of focused ion beam (FIB; Nova 600 NanoLab, Dual Beam SEM/FIB, FEI, The Netherlands), using 30 kV Ga ions for milling and 5 kV for the final cleaning step.

Table 2. Overview of the tested materials and their corresponding exposure conditions.

Material	Parasitic Phases (XRD Data)	Material Ref.	T (°C)	Time (h)	Plot in Fig. 2	Flow Condition
Maxthal® 312 (Ti ₃ SiC ₂)	TiC	Kanthal AB, Sweden [31]	500	3500	(1)	Static
Maxthal® 211 (Ti ₂ AlC + Ti ₃ AlC ₂)	TiAl ₃	Kanthal AB, Sweden [31]	500	3500	(1)	Static
(Ti,Nb)₂AlC	TiAl ₃ , Al ₂ O ₃	/	500	3500	(1)	Static
Ti₃AlC₂	/	/	500	3500	(1)	Static
Nb₂AlC	Al ₂ O ₃	/	500	3500	(1)	Static
Nb₄AlC₃	NbAl ₃ , Al ₂ O ₃	/	500	3500	(1)	Static
(Nb_{0.85},Zr_{0.15})₄AlC₃ (*)	Al ₂ O ₃	[32]	500	1000	(2)	Static
			500	1000	(3)	v ≈ 8 m/s
Zr₂AlC	ZrC	[33]	500	1000	(2)	Static
			500	1000	(3)	v ≈ 8 m/s
Zr₃AlC₂	ZrC, ZrAl ₂	[34]	500	1000	(2)	Static
			500	1000	(3)	v ≈ 8 m/s

(Zr_{0.8},Ti_{0.2})₂AlC & (Zr_{0.8},Ti_{0.2})₃AlC₂	(Zr,Ti)C	[35]	500	1000	(2)	Static
Ti₂SnC	Sn, TiC	[25]	500	1000	(4)	Static
316L SA & 316L CW	/	[6]	500	1000/3282	/	Static
316L SA	/	[6]	500	1000	(3)	v ≈ 8 m/s

^(*) Two surface conditions were tested in the CORELLA, i.e., ground and conventionally milled; SA = solution-annealed; CW = cold-worked

3. Results and Discussion

3.1. Exposures to Static LBE

An overview of the microstructure of the pristine MAX phase ceramics is provided in Fig. 3. The constituent phases are indicated on the micrographs and listed in Table 2. In most grades, the MAX phase grains can be easily identified by their lamellar shape, which reflects their hexagonal crystal structure with large c/a ratio and with the c -axis oriented perpendicular to the longer direction. The backscattered electron (BSE) images of Fig. 3 indicate that parasitic phases were present in nearly all MAX phase-based ceramics. Only in Ti_3AlC_2 , no evidence of another phase was found. Three types of parasitic phases could be identified: oxides, (pseudo-)binary carbides and A-rich compounds. Alumina (Al_2O_3) particles were detected in all Nb-containing materials and resulted from the aluminothermal reduction of Nb_2O_5 , which was an impurity in the Nb starting powder. Al_2O_3 particles were small ($<5\text{ }\mu\text{m}$) and well-dispersed in the material microstructure: they were present at grain boundaries or inside the grains, as in the case of the $(\text{Ti,Nb})_2\text{AlC}$ and $(\text{Nb}_{0.85},\text{Zr}_{0.15})_4\text{AlC}_3$ ceramics (Fig. 3). These small oxide grains did not jeopardize the material's machinability, as demonstrated for milled $(\text{Nb}_{0.85},\text{Zr}_{0.15})_4\text{AlC}_3$. Moreover, they have been reported to improve the flexural strength and fracture toughness of MAX phase ceramics [36]. The (pseudo-)binary carbides were present in Maxthal[®] 312 (TiC), in the Zr-rich ceramics (ZrC and (Zr,Ti)C) and in Ti_2SnC (TiC). These carbides were the main competing phases during MAX phase synthesis and their complete elimination was challenging, especially in the case of the Zr-rich MAX phases. The carbide grains were very fine (in the micrometre to sub-micrometre range for (Zr,Ti)C) and preferred to cluster together between the MAX lamellae. The third group of parasitic phases comprised A-rich compounds. These were typically intermetallics, i.e., TiAl , NbAl_3 and ZrAl_2 , or occasionally metallic A, such as Sn, in the case of Ti_2SnC . These phases were mainly present at grain boundaries and in triple points. They have been reported to be most susceptible to liquid metal attack and, hence, are considered as most detrimental for the corrosion resistance of MAX phase-based materials [15]. This statement was confirmed for the 11 materials exposed to static liquid LBE in this work.

An overview of the 11 MAX phase ceramics after exposure to static LBE is given in Fig. 4. The BSE images show the MAX/LBE interface at the mirror-polished side. The bright phase on top of all materials are solidified LBE residues that create a strong compositional contrast with the MAX phase materials due to large differences in the respective mean atomic numbers. A first observation is that all polished surfaces remained intact, leaving the MAX/LBE interfaces flat. This is in stark contrast with the dissolution corrosion damages observed in 316L stainless steels exposed to similar test conditions. Examples of such damages are presented in Fig. 5 for two 316L steel heats that were previously exposed by Lambrinou et al. [6] to oxygen-poor ($C_o < 2.2 \times 10^{-8}$ mass%), static liquid LBE, at 500°C , for 1000 h and 3282 h. The two steel heats were: one solution-annealed heat (here designated 316L SA; also exposed to fast-flowing LBE in this work) and a cold-worked heat (here designated 316L CW; described as 316LH1 in Ref. [6]). Both 316L steel heats are included in the list of tested materials presented in Table 2. As shown in Fig. 5, dissolution corrosion damages in 316L stainless steels exposed to comparable conditions

are much more severe, especially for cold-worked steels (reaching locally $\sim 350\text{ }\mu\text{m}$ in the 316L CW steel), than in the MAX phase-based ceramics. Moreover, LBE penetrates into 316L stainless steels by following preferred ingress paths in the steel microstructure (e.g., grain boundaries, deformation twin boundaries, etc.) and the steel/LBE interaction is catastrophic, i.e., the LBE-affected zones lose their load-bearing capacity. The dissolution corrosion behaviour of 316L stainless steels has been previously addressed by Lambrinou et al. [6] and does not merit further analysis here, except for comparison with the behaviour of the MAX phases. In contrast with the adverse effects of LBE dissolution attack on 316L steels, the LBE penetration into the MAX phases occurs in an orderly manner, without destroying the MAX phase crystal structure and, presumably, without severe – if any – loss in load-bearing capacity. Even though the latter statement should still be confirmed experimentally, this work provides ample evidence that the MAX phase crystal structure is preserved during the observed LBE ingress into Zr-based MAX phases (the latter being the only MAX phase compounds that showed interaction with liquid LBE). The interaction of Zr-based MAX phases with oxygen-poor, static LBE will be extensively addressed later in the text.

The following materials did not show any sign of interaction with the liquid LBE: Maxthal[®] 312, Maxthal[®] 211, $(\text{Ti,Nb})_2\text{AlC}$, Nb_2AlC , Nb_4AlC_3 and $(\text{Nb}_{0.85},\text{Zr}_{0.15})_4\text{AlC}_3$. No LBE traces were detected in the material bulk for these grades, and any impression of sub-surface LBE presence in some of these materials, i.e., in Maxthal[®] 211 and $(\text{Ti,Nb})_2\text{AlC}$ (Fig. 4), can be attributed to local LBE smearing during polishing of the metallographic cross-sections. Moreover, the very low LBE C_0 during these exposures did not form an oxide scale resolvable by SEM; this could be attributed to one of the following two reasons: either these MAX phase ceramics were intrinsically stable to LBE dissolution attack or they were protected by a nanometric protective oxide film covering the specimen surface. For example, Al_2O_3 and SiO_2 thin films could be protective at the reported exposure conditions, as both Al_2O_3 and SiO_2 are thermodynamically stable at $C_0 < 10^{-15}$ mass% at 500°C in liquid LBE [37]. In order to examine whether the presence of a nanometric (not resolvable by SEM), protective oxide film prevented the occurrence of LBE dissolution attack, FIB/TEM/EDS work was performed on non-attacked by LBE areas of the following MAX phase ceramics: Zr_2AlC , $(\text{Zr}_{0.8},\text{Ti}_{0.2})_{n+1}\text{AlC}_n$, $(\text{Nb}_{0.85},\text{Zr}_{0.15})_4\text{AlC}_3$ and Maxthal[®] 211 (nominally, Ti_2AlC). Specimen selection for this detailed work aimed at providing an explanation for the good compatibility of MAX phase compounds in the (Zr,Ti,Nb)-Al-C system with oxygen-poor liquid LBE at 500°C .

For the remaining ceramics, two degrees of interaction could be distinguished: the weakest interaction was observed for Ti_3AlC_2 and Ti_2SnC , with the LBE penetrating locally into grain boundaries. For Ti_3AlC_2 , the interaction was very local with a maximum depth of LBE penetration of $\sim 20\text{ }\mu\text{m}$, while both the bulk of the MAX phase grains and the overall material integrity were not affected. A stronger interaction was observed for Ti_2SnC : since Sn melts at 232°C , this minor phase in the material was removed, dissolving into the liquid LBE. The homogeneous presence of Pb and Bi in the specimen suggests that liquid LBE percolated through the interconnected network of Sn-rich areas into the material bulk, leaving the Ti_2SnC MAX phase grains intact. For both Ti_3AlC_2 and Ti_2SnC , the type of observed interaction is similar to that reported for Ti_2AlC

by Heinzl et al. [15], i.e., the intergranular LBE penetration resulted in LBE dissolution attack of the A-rich phase (TiAl_3), while the MAX phase grains remained unaffected. Differences in the liquid metal corrosion behaviour of the MAX phases and their (often unavoidable) parasitic phases is an additional reason for targeting high phase purity in MAX phase-based materials intended for HLM-cooled nuclear systems.

The most severe interaction was observed in the Zr-rich MAX phase ceramics Zr_2AlC , Zr_3AlC_2 and $(\text{Zr}_{0.8}, \text{Ti}_{0.2})_{n+1}\text{AlC}_n$. In these materials, the intermetallics at grain boundaries underwent LBE dissolution attack and even the MAX phase grains were affected by the HLM. The bright zones in these materials correspond to LBE-affected regions and can be easily distinguished from the unaffected (pseudo-)binary carbides, as illustrated in Fig. 6b for ZrC in Zr_2AlC and in Fig. 9a for (Zr,Ti)C in $(\text{Zr}_{0.8}, \text{Ti}_{0.2})_{n+1}\text{AlC}_n$. Despite the observed MAX/LBE interaction, the specimen surface appeared intact and the specimens kept their structural integrity. No deterioration similar to that observed for Ti_3AlC_2 in liquid sodium (Na) was observed [21]. Moreover, the LBE-affected zone did not have uniform thickness and the LBE penetration depth could exceed locally 100 μm , whereas no interaction was observed in adjacent regions, as shown in Fig. 6a for the Zr_2AlC -based ceramic. In an attempt to explain such localised differences in the corrosion behaviour of the Zr_2AlC -based MAX phase ceramic, a FIB foil was extracted from a non-affected material area and studied by TEM/STEM/EDS, and the results of this dedicated study will be discussed later in the text.

The propagation of the MAX/LBE interaction front is rather unusual: as shown for Zr_2AlC , the LBE penetration into the MAX phase grains occurs in a layered manner, resulting in a stripy grain pattern (Fig. 6c). Considering the nanolaminated crystal structure of the MAX phases, the planes of LBE penetration are most likely A-layers where Al is partially replaced by Pb and/or Bi. Apart from the easy to understand LBE diffusion along the basal planes, LBE also diffuses almost perpendicular to the basal planes inside the MAX phase grains, as shown by the LBE-rich protrusions observed in a grain on the right hand-side of Fig. 6c (pinpointed by arrow). The diffusion of Pb and Bi away from the basal planes in Zr_2AlC is also confirmed by the EDS elemental maps in Fig. 7a. The Al-atoms that leave the MAX phase structure tend to segregate at grain boundaries, as shown by the Al-decorated grain boundaries of LBE-affected Zr_2AlC MAX phase grains in Fig. 7b. In this context, the work of Horlait et al. [38, 39] is of great interest: they investigated the possibility to stabilize the Zr_2AlC -structure by producing M- and A-site solid solutions, showing that both Pb and Bi could be introduced on the A-site, forming $\text{Zr}_2(\text{Al}_{0.35}, \text{Pb}_{0.65})\text{C}$ and $\text{Zr}_2(\text{Al}_{0.42}, \text{Bi}_{0.58})\text{C}$, respectively. The Zr_2PbC end-member is known since the 1960s [40], but it was not possible to synthesise Zr_2BiC [39]. Moreover, the attempt to synthesise $\text{Zr}_2(\text{Pb}, \text{Bi})\text{C}$ with a eutectic Pb:Bi ratio (atomic ratio $\approx 45:55$) proved unsuccessful. In fact, Bi could not be incorporated in the MAX phase structure, and only Zr_2PbC was produced [38]. In the present work, all three A-elements were detected in the LBE-affected MAX phase lamellae, suggesting the synthesis of new $\text{Zr}_{n+1}(\text{Al}, \text{Pb}, \text{Bi})\text{C}_n$ and $(\text{Zr}_{0.8}, \text{Ti}_{0.2})_{n+1}(\text{Al}, \text{Pb}, \text{Bi})\text{C}_n$ MAX phase solid solutions.

In order to verify whether the MAX phase structure was maintained over large parts of the LBE-affected zones, EBSD was employed, as shown in Fig. 6d for the Zr_2AlC -based ceramic. The use of EBSD in conjunction with EDS elemental mapping of the LBE-affected zone (Fig. 6e) showed that LBE-affected grains could still be indexed as the Zr_2AlC MAX phase compound. In order to confirm beyond any doubt the retention of the MAX phase crystal structure, despite the incorporation of Pb/Bi atoms, SAED was employed on FIB foils lifted out of the LBE-affected zone. The proof of the retention of the MAX phase crystal structure is provided in Fig. 6f, which confirms that a MAX phase grain in the centre of the LBE-affected zone of the Zr_2AlC -based ceramic has indeed the Zr_2AlC crystal structure (the SAED pattern shown in Fig. 6f is the $2\bar{1}\bar{1}0$ zone axis of Zr_2AlC). The lattice parameters a and c of the $\text{Zr}_2(\text{Al,Pb,Bi})\text{C}$ solid solution are estimated based on SAED patterns from 7 grains in the LBE-affected zone and are compared to those of related Zr-based MAX phase compounds in Table 3. In comparison with Zr_2AlC , the lattice expanded significantly due to the incorporation of Pb and Bi atoms. This can be attributed to the larger atomic radius of these two atoms as compared to Al [39]. The expansion along the a direction is comparable to that observed for the Bi-containing solid solution $\text{Zr}_2(\text{Al}_{0.42},\text{Bi}_{0.58})\text{C}$ [39] and Zr_2PbC [41]. On average, the lattice expands more in the c direction (2.5% as compared to Zr_2AlC) and the c -axis is notably larger than in any other reported 211 compound [42]. The retention of the MAX phase crystal structure in LBE-affected areas was also confirmed by SAED and STEM/EDS for the exposed $(\text{Zr}_{0.8},\text{Ti}_{0.2})_{n+1}\text{AlC}_n$ ceramic: the presence of Pb/Bi in both 211 and 312 MAX phase grains in this material (Fig. 9d) did not alter the crystal structure (the SAED patterns in Fig. 9b show the $2\bar{1}\bar{1}0$ zone axis of the 211 and 312 MAX phases for two of the LBE-affected grains).

Table 3. Lattice parameters (LPs) for $\text{Zr}_2(\text{Al,Pb,Bi})\text{C}$ and related MAX phase compounds.

LP	$\text{Zr}_2(\text{Al}_{0.65},\text{Pb}_{0.16},\text{Bi}_{0.19})\text{C}$	Zr_2AlC [33]	Zr_2PbC [41]	$\text{Zr}_2(\text{Al}_{0.42},\text{Bi}_{0.58})\text{C}$ [39]
a (Å)	3.37 ± 0.03	$3.32 \pm 2 \times 10^{-4}$	3.38	$3.34 \pm 5 \times 10^{-3}$
c (Å)	14.93 ± 0.35	$14.57 \pm 4 \times 10^{-4}$	14.67	$14.51 \pm 2 \times 10^{-2}$

The Zr_2AlC ceramic showed the deepest LBE penetration ($\sim 150 \mu\text{m}$); the latter was mainly, but not exclusively, concentrated at specimen corners, suggesting that the local concentration of (presumably tensile) stresses at these sites might have facilitated the LBE ingress into the bulk. The Zr_2AlC /LBE interaction front appeared rather diffuse and was characterised by a gradual increase in the Pb and Bi contents that reached a maximum in the LBE-affected zone. An EPMA elemental mapping of the transition zone is shown in Fig. 7c. The Zr-map shows an apparent decrease in Zr intensity throughout the Pb/Bi-containing zone. In order to verify the possible loss of Zr, the $\text{Zr}:(\text{Al}+\text{Pb}+\text{Bi})$ ratio was compared to the $\text{Zr}:\text{Al}$ ratio in the unaffected zone, revealing a ratio of 65:35 and 67:33, respectively. This difference is small and close to the statistical error of the method; moreover, the measured difference in the Zr intensity can be partially attributed to the reduced electron and X-ray interaction volume due to the presence

of the two heavy elements (Pb, Bi). Hence, EPMA does not provide concrete evidence of the suspected partial substitution of Zr by Pb and/or Bi in Zr_2AlC .

The Al:(Pb+Bi) ratio in the zones that converted fully into the $\text{Zr}_2(\text{Al,Pb,Bi})\text{C}$ solid solution was determined to be 65:35, implying that Al remained the dominant A-element, while no further Al gradient was observed towards the sample surface. The Pb:Bi ratio was 47:53, which is close to the eutectic LBE ratio of 45:55, suggesting no preferential replacement of Al by Pb or Bi. The partial substitution of A-atoms resembles to a certain extent the synthesis technique recently employed to produce Ti_3AuC_2 [43]. Fashandi et al. heat-treated Au-coated Ti_3SiC_2 for 12 h at 670°C , resulting in the complete replacement of the A-element (Si) by Au-atoms that was also accompanied by the elongation of the *c*-axis by 5.6% [43]. In the present work, approximately 1/3 of the Al atoms were replaced by Pb and Bi, and the resulting expansion along the *c*-axis was also considerable. The Al:(Pb+Bi) ratio was constant across the fully converted material volume, indicating an upper limit in the amount of Al that can be substituted by Pb and Bi at 500°C . Additional experiments are required to find out whether the Al:(Pb+Bi) $\approx$ 2:1 ratio in the $\text{Zr}_2(\text{Al,Pb,Bi})\text{C}$ solid solution is temperature-dependent, which would suggest that this atomic ratio corresponds to the solid-solubility limit of Pb+Bi in the Zr_2AlC MAX phase.

The other Zr-Al-C grade, Zr_3AlC_2 , showed significantly less LBE penetration into the bulk, which was limited to few localized spots. This indicates that the higher order (312) Zr_3AlC_2 MAX phase compound is chemically more compatible with liquid LBE at 500°C than its lower order (211) Zr_2AlC counterpart. This is also exemplified in $(\text{Zr}_{0.8},\text{Ti}_{0.2})_{n+1}\text{AlC}_n$: this ceramic was a mixture of 211 and 312 phases in the as-processed state, where the sandwich-like MAX phase grains were made of a brighter 312-rich core enveloped by a darker 211-based outer layer (Fig. 3). After exposure, this phase contrast was inverted (Figs. 4 and 9a) due to differences in the degree of interaction of the two MAX phase grain constituents with LBE, resulting in sandwich-like grains with a brighter, LBE-rich (Al:(Pb+Bi) $\approx$ 76:24) outer layer and a darker, LBE-poorer (Al:(Pb+Bi) $\approx$ 82:18) core.

The TEM/STEM/EDS analysis of FIB foils extracted from seemingly unaffected by LBE areas in select MAX phase ceramics yielded interesting, material-specific results. First, the study of a random location (*x-x'* in Fig. 8a) in an unaffected area of the Zr_2AlC surface revealed the sub-surface segregation of ZrC particles (Fig. 8b). As observed in the LBE-affected zones of Zr_2AlC (Fig. 6b), ZrC is impervious to LBE dissolution attack, appearing as unaffected ZrC 'islands' in a matrix of $\text{Zr}_2(\text{Al,Pb,Bi})\text{C}$ laths. Hence, ZrC particle segregation in parts of the specimen surface protected locally the underlying MAX phase grains from LBE ingress, at least within the 1000-h timeframe of the performed exposure. The formation of ZrC was impossible to suppress during processing, thereby becoming the main competing phase in the Zr_2AlC -based ceramic [33]. The destabilisation of the Zr_2AlC phase in this ceramic yielded local segregations of ZrC and Zr-Al intermetallics (typically, ZrAl_3 , but the formation of ZrAl_2 could also not be excluded [33]). EDS elemental mapping of such segregations of parasitic phases in the bulk of the Zr_2AlC ceramic confirmed the coexistence of ZrC grains with intergranular Zr-Al intermetallics (IMCs), even though XRD had detected ZrC as the only parasitic phase (Fig. 8c). The exact composition

of these IMCs could not be determined by EDS, due to the fine grain size ($<1\ \mu\text{m}$) of these aggregates. As shown in Fig. 8c, LBE percolated through the cluster of ZrC particles, using the grain boundaries as ingress path; hence, one could expect that it is only a matter of time before LBE goes completely through such aggregates, affecting the underlying MAX phase grains. Moreover, LBE seemed to affect the Zr-Al IMCs, while leaving ZrC completely intact. The greater IMC susceptibility to LBE dissolution attack was also confirmed in the $(\text{Zr}_{0.8}, \text{Ti}_{0.2})_{n+1}\text{AlC}_n$ ceramic: the (Zr,Ti)-Al IMCs present between the (Zr,Ti)C grains close to the exposed specimen surface were fully dissolved by LBE (Fig. 9c). This finding is in agreement with the work of Heinzl et al. [15], who reported preferential LBE dissolution attack on the parasitic phases (primarily, Ti-Al IMCs) of Maxthal® 211 (nominally, Ti_2AlC) that had been exposed to oxygen-containing ($C_O \approx 10^{-8}$ mass%) static liquid LBE at 650°C for 10,000 h.

Second, the study of a random location (x-x' in Fig. 10a) on the $(\text{Nb}_{0.85}, \text{Zr}_{0.15})_4\text{AlC}_3$ specimen surface revealed the formation of a thin (typically, $<1\ \mu\text{m}$; maximum local thickness $\sim 1.4\ \mu\text{m}$), seemingly discontinuous oxide scale. Analysis of this oxide scale at a site of maximum thickness (Fig. 10b) showed it to be Al-rich (Fig. 10d) and in the vitreous (amorphous) state (Fig. 10c). In fact, this MAX phase ceramic formed continuous Nb-based (Nb_2O_5) oxide scales upon its exposure to oxygen-containing ($C_O \approx 5 \times 10^{-9}$ mass%), fast-flowing liquid LBE at 500°C (see section 3.2). Hence, the formation of a thin and discontinuous (Al-based) oxide scale as result of its exposure to oxygen-poor ($C_O < 10^{-10}$ mass%) static LBE at 500°C indicates that the oxygen potential was not sufficient to form a Nb-based oxide, but formed an Al-based thin film instead, since Al_2O_3 is stable at very low oxygen potentials ($C_O < 10^{-15}$ mass% at 500°C in LBE [37]). The absence of a continuous, protective oxide scale on the material surface, however, suggests that it is inherently resistant to dissolution corrosion at the considered exposure conditions.

Third, the study of a random location (x-x' in Fig. 11a) on the Maxthal® 211 specimen surface revealed the presence of an oxide scale with non-uniform thickness (typically, $<0.5\ \mu\text{m}$; locally, $>2.5\ \mu\text{m}$; Fig. 11b). In the areas where the oxide scale was thinnest, the oxide scale appeared to be Ti-based, while in areas of enhanced local thickness, it appeared to be Al-rich (Fig. 11c). The careful consideration of the EDS elemental maps of Fig. 11c suggests that the thicker (Al-based or mixed Al-Ti oxides) oxide scales resulted from the oxidation of the Ti-Al IMCs that are present between the MAX phase grains. On the other hand, the thinner (Ti-based, presumably TiO_2) oxide scales resulted from the oxidation of Ti_2AlC . The formation of TiO_2 (rutile) oxide scales on Maxthal® 211 (nominally, Ti_2AlC ; usually, mixture of Ti_2AlC , Ti_3AlC_2 and TiAl_3 – see Table 2) exposed to liquid LBE has been previously reported by Heinzl et al. [15]. Moreover, the same authors observed the preferential oxidation of the parasitic phases (mainly, Ti-Al IMCs) in Maxthal® 211 exposed to oxygen-containing ($C_O \approx 10^{-6}$ mass%) static liquid LBE at 550°C for up to 10,000 h [15].

In conclusion, the screening exposures of select MAX phases to oxygen-poor static liquid LBE showed a satisfactory overall compatibility with the HLM, except from a limited local interaction in the Zr_2AlC , Zr_3AlC_2 and $(\text{Zr}_{0.8}, \text{Ti}_{0.2})_{n+1}\text{AlC}_n$ ceramics. The local interaction between LBE and Zr-based MAX phases formed in-situ Pb/Bi-containing MAX phase solid solutions, a phenomenon meriting further in-depth investigation to be fully understood. Since the formation of Pb/Bi-

containing solid solutions occurred without the dissociation of the exposed Zr-based MAX phase compounds, one might assume that the LBE-affected parts of Zr-based ceramics will not lose completely their load-bearing capacity. This is in stark contrast with components made of 316L austenitic stainless steels, where the dissolution-affected parts lose structural integrity in a dramatic and irreversible manner. The in-depth understanding of the liquid metal corrosion behaviour of MAX phase ceramics in contact with HLMS, e.g., LBE and Pb, can only be achieved by the systematic testing of these materials in different exposure conditions (T , C_O , time), followed by detailed post-exposure material characterisation. This study showed a material-specific corrosion resistance that was greatly affected by the performance of parasitic phases. This underlines the importance of phase purity for MAX phase ceramics intended for specific applications in HLM-cooled nuclear systems.

3.2. Exposure to Fast-Flowing LBE

Select MAX phase-based ceramics, i.e., $(\text{Nb}_{0.85}\text{Zr}_{0.15})_4\text{AlC}_3$ (NZ413), Zr_2AlC and Zr_3AlC_2 , were exposed to oxygen-containing ($C_O \approx 5 \times 10^{-9}$ mass%), fast-flowing ($v \approx 8$ m/s) liquid LBE at 500°C for 1000 h. Two different surface finishes of the NZ413 grade, i.e., ground and conventionally milled, were exposed simultaneously to study the effect of surface finish on the development of erosion damages. A (ground) 316L austenitic stainless steel specimen was included in the same test, as reference structural material; this specimen was made of the 316L SA solution-annealed heat (Table 2). An overview of the bulk pristine and tested materials is presented in Fig. 12, together with the surface topography of the specimens before and after exposure. Fig. 12 also provides a comparison of the surface finishes achieved by diamond wheel grinding and conventional milling for the NZ413 grade. The measured average roughness, S_a , and the maximum roughness, S_z , values for the 5 pristine and exposed specimens are given in Table 4. Changes in the material surface roughness provide a good indication of the material loss due to erosion phenomena resulting from exposure to fast-flowing liquid LBE. Fig. 12 shows that both Zr-Al-C MAX phases broke at the clamping point upon their removal from the test facility. This could be primarily attributed to the non-negligible amount of the brittle ZrC parasitic phase that was present in both MAX phase ceramics.

Table 4. Surface roughness values before and after material exposure to fast-flowing LBE.

		Zr₂AlC	Zr₃AlC₂	Ground NZ413	Milled NZ413	316L SA
Before exposure	S_a (μm)	0.37	0.35	0.30	2.12	0.80
	S_z (μm)	3.97	3.90	3.42	21.37	15.37
After exposure	S_a (μm)	0.79	10.80	0.64	2.85	6.43
	S_z (μm)	14.51	104.80	4.83	23.90	75.97

The colour change and strong increase in surface roughness of Zr_3AlC_2 after exposure (Fig. 12) was attributed to severe oxidation. The ZrO_2 -based oxide scale that covered the specimen surface was quite porous and poorly adherent (Fig. 13b), i.e., it is expected to be incapable of protecting the substrate ceramic from further oxidation. A similar but milder oxidation was observed for Zr_2AlC (Fig. 13a). No LBE penetration into the grains of both Zr-Al-C MAX phase ceramics was detected, as opposed to their exposure to static LBE. Such difference in corrosion behaviour can be associated to differences in the exposure conditions: first, the LBE oxygen concentration was sufficient to promote oxidation in contact with moderately oxygenated ($C_O \approx 5 \times 10^{-9}$ mass%) fast-flowing LBE, while suppressing it in contact with oxygen-poor ($C_O < 10^{-10}$ mass%) static LBE. Moreover, the missing data from the first 300 h of exposure in the CORELLA do not allow a precise assessment of the effect of the initial LBE oxygen concentration on the material corrosion behaviour. Unlike the exposure to static LBE, the fast LBE flow ($v \approx 8$ m/s) ensured the continuous and uniform mass transport of oxygen to the material surface, thus promoting oxide scale growth. The herein described exposure conditions resulted in oxidation as the prevailing corrosion mechanism, and despite the fact that the observed oxide scales were porous and not perfectly protective (e.g., LBE was detected locally in the oxide scale), the LBE ingress into the MAX phase grains was prevented, at least within the 1000-h timeframe of the CORELLA test. It should be noted that the enhanced surface roughness (Table 4) caused by oxidation made it difficult to accurately determine erosion damages.

The formation of a thin ($\sim 3\text{-}4$ μm) oxide scale on the surface of the two NZ413 grades (Figs. 13c and 13g) is not surprising, taking into account that Nb-based MAX phases have been reported to be prone to oxidation above 500°C in air [44]. The observed Nb_2O_5 -based oxide scale was continuous and rather defective, especially in the case of the milled NZ413 (Fig. 13c). By contrast, a discontinuous, Al-based oxide scale formed on the surface of this ceramic during its exposure for the same amount of time (1000 h) to oxygen-poor static LBE; this can be associated with the extremely limited and non-uniform (due to the absence of mixing) oxygen supply to the material's surface under these test conditions. Apart from the thin oxide scale, no other corrosion/erosion damages were observed on the NZ413 MAX phase grades. Even the characteristic surface topology of the milled NZ413 was retained in a quasi-identical state in the exposed ceramic, while the ground surface was also found intact (Fig. 12). This indicates negligible – if any – material loss during the exposure of NZ413 to fast-flowing LBE, in strong contrast with the observed erosion damages on the 316L stainless steel as result of the same exposure. The latter is confirmed in Figs. 13d-13g, which compare the post-exposure surface roughness of ground NZ413 MAX phase and 316L stainless steel specimens: the steel shows appreciable material loss, as opposed to NZ413 that appears intact. The poor performance of the 316L structural steel is similar to that reported by Müller et al. [45] for a 316L stainless steel exposed to oxygen-containing ($C_O \approx 10^{-6}$ mass%) flowing LBE ($v \approx 2$ m/s) at 600°C for 2000 h. Under such conditions, the formation of a protective oxide scale was suppressed and the 316L steel disintegrated by massive ablation of the dissolution-affected zone. Similar exposure conditions (i.e., $T = 500^\circ\text{C}$, $C_O \leq 10^{-8}$ mass%, 1000 h) to the herein reported ones have been previously reported by Lambrinou et al. [6] to promote LBE dissolution attack on the same 316L

steel heat (i.e., 316L SA) in contact with static LBE. The high LBE flow velocity ($v \approx 8$ m/s) in the CORELLA test is expected to have further undermined the formation of a continuous, protective oxide scale on the steel surface and to have removed the dissolution-affected material areas, as previously observed by Müller et al. [45]. Based on these observations, it is clear that solution-annealed 316L steels are not ideal structural materials for similar service conditions.

In conclusion, the screening exposure of select MAX phase ceramics (i.e., 3 of the 11 materials initially exposed to oxygen-poor, static LBE) to oxygen-containing, fast-flowing LBE at 500°C showed that NZ413 was the most suitable candidate material for use in conditions relevant to the pump impeller application. The findings of this work suggest that this material could remain stable in other Pb-alloys (e.g., Pb, Pb-17%Li, Pb-16%Au, etc.) at comparable (HLM-specific) exposure temperatures, on the condition that the HLM C_O is sufficiently low. Systematic work is still needed to define the envelop of safe service conditions for this and other promising MAX phase-based materials.

4. Conclusions

In this work, different MAX phase-based ceramics were exposed to oxygen-poor static and fast-flowing liquid LBE at 500°C to assess their performance in conditions relevant to the harsh service conditions of pump impeller components in Gen-IV LFRs. First, 11 different ceramics were exposed to oxygen-poor ($C_O \leq 2.2 \times 10^{-10}$ mass%) static liquid LBE at 500°C for up to 3500 h to evaluate their intrinsic resistance to LBE dissolution attack. The main findings of this part of the study could be summarised as follows:

1. The compatibility of the MAX phase-based ceramics with liquid LBE was, in general, good, without any signs of structural disintegration of any of the exposed ceramics. Material performance was affected by the presence of parasitic phases, in particular intermetallics and low-melting point (e.g., Sn) metallic inclusions, indicating that phase purity is an essential requirement for MAX phase ceramics intended for use in HLM-cooled nuclear systems.
2. The low LBE oxygen concentration suppressed the formation of continuous, defect-free protective oxide scales. The superb resistance of specific MAX phase-based materials (e.g., $(\text{Nb}_{0.85}, \text{Zr}_{0.15})_4\text{AlC}_3$, Maxthal® 211) to LBE dissolution attack suggests that they are chemically compatible with oxygen-poor LBE at high temperatures, unlike the reference 316L stainless steels that suffer severe corrosion damages, often locally enhanced.
3. A mild LBE interaction was observed for Ti_2SnC , due to the presence of a minor amount of parasitic Sn, which facilitated the LBE ingress due to the replacement of Sn by Pb/Bi.
4. The strongest LBE interaction was observed for the Zr-rich MAX phase-based ceramics Zr_2AlC , Zr_3AlC_2 and $(\text{Zr}_{0.8}, \text{Ti}_{0.2})_{n+1}\text{AlC}_n$. In general, Pb/Bi atoms were incorporated in the MAX phase crystal structure, resulting in an expansion of the lattice, especially along the *c*-axis. The LBE/MAX phase interaction resulted in the in-situ formation of new MAX phase solid solutions $\text{Zr}_2(\text{Al}, \text{Pb}, \text{Bi})\text{C}$ and $(\text{Zr}_{0.8}, \text{Ti}_{0.2})_{n+1}\text{AlC}_n$. Such LBE/MAX interaction is not expected to reduce the load-bearing capacity of the affected MAX phase ceramics, unlike the compromising dissolution corrosion damages observed in 316L steels.

In a second step, 3 of the 11 MAX phase ceramics were exposed to oxygen-poor ($C_O \approx 5 \times 10^{-9}$ mass%) fast-flowing ($v \approx 8$ m/s) liquid LBE at 500°C for 1000 h to evaluate their resistance to erosion. The main findings of this part of the study could be summarised as follows:

1. None of the MAX phase-based ceramics showed clear signs of erosion damage. On the contrary, the reference 316L SA stainless steel exhibited appreciable mass loss, due to flow-assisted corrosion (erosion) effects.
2. Despite the rather low LBE oxygen concentration, oxidation was the prevailing corrosion mechanism, especially for the Zr_3AlC_2 and Zr_2AlC grades. The controlled oxidation of the $(Nb_{0.85},Zr_{0.15})_4AlC_3$ grade formed a relatively thin, continuous Nb_2O_5 oxide scale, irrespective of the surface finish (i.e., ground vs. conventionally milled).
3. The $(Nb_{0.85},Zr_{0.15})_4AlC_3$ grade was the most stable of the 3 tested MAX phase ceramics in contact with fast-flowing, oxygen-poor liquid LBE, suggesting that this might be a promising candidate structural material for the pump impeller application, at least for parts expected to experience severe erosion damages (e.g., the rotor blades).

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Fig. 1. (a) Experimental setup used to expose MAX phase ceramics to oxygen-poor, static LBE. One of the graphite plates used to immerse the MAX phase ceramics in the LBE bath prior to (b) and after (c) the 3500 h-long exposure to oxygen-poor ($C_o < 10^{-10}$ mass%) liquid LBE at 500°C.

Fig. 2. Evolution of the dissolved oxygen concentration in liquid LBE during the exposures performed in this work. Plots (1), (2) and (4) refer to tests in static LBE, and plot (3) to testing in fast-flowing LBE.

Fig. 3. Backscattered electron (BSE) images of the pristine MAX phase ceramics, indicating all parasitic phases.

Fig. 4. BSE images of the LBE/MAX phase interaction for all MAX phase ceramics exposed to static LBE for different times.

Fig. 5. BSE images showing dissolution corrosion damages observed in 316L stainless steels exposed to oxygen-poor ($C_O \leq 10^{-8}$ mass%) static LBE, at 500°C for 1000-3282 h: (a,c) 316L SA steel; (b,d) 316L CW steel.

Fig. 6. BSE images of the Zr_2AlC -based ceramic exposed for 1000 h to oxygen-poor, static LBE: (a) non-uniform attack, showing LBE-affected areas next to seemingly unaffected areas, (b) area in the centre of the LBE-affected zone (site I in Fig. 6a), (c) area at the Zr_2AlC /LBE interaction front (site II in Fig. 6a). (d) EBSD grain orientation map and (e) respective EDS Bi map of a location close to the Zr_2AlC /LBE interaction front. (f) SAED pattern showing the preservation of the MAX phase crystal structure in the LBE-affected zone of Zr_2AlC .

Fig. 7. (a) EDS elemental maps showing Pb/Bi diffusion away from the basal planes in the Zr₂AlC-based ceramic. (b) EDS elemental maps of Zr₂(Al,Pb,Bi)C MAX phase grains: Al-rich grain boundaries and chemically inert ZrC grains. (c) EPMA elemental mapping close to the Zr₂AlC/LBE interaction front.

Fig. 8. (a) BSE image of a non-attacked area in the Zr₂AlC-based ceramic exposed to 1000 h to oxygen-poor, static LBE. (b) FIB cross-section along x-x' in Fig. 8a and EDS elemental maps showing segregation of ZrC particles at the specimen surface. (c) Segregation of ZrC particles in an LBE-affected zone: LBE penetrates intergranularly in this biphasic area (made of ZrC and Zr-Al IMCs), leaving ZrC intact.

Fig. 9. (a) BSE image of the $(\text{Zr}_{0.8}\text{Ti}_{0.2})_{n+1}\text{AlC}_n$ -based ceramic exposed for 1000 h to oxygen-poor, static LBE. (b) HAADF STEM image of an area containing 312 and 211 MAX phase and $(\text{Zr,Ti})\text{C}$ grains, along with the SAED patterns showing the $[2\bar{1}\bar{1}0]$ zone axes of both MAX phases. (c) HAADF STEM image of the area at the upper right corner of Fig. 9b: LBE penetration into $(\text{Zr,Ti})\text{C}$ grain boundaries and LBE dissolution attack of the Zr-Al IMCs. (d) STEM EDS elemental maps of the whole area shown in Fig. 9b.

Fig. 10. (a) BSE image of the $(\text{Nb}_{0.85}\text{Zr}_{0.15})_4\text{AlC}_3$ -based ceramic exposed for 1000 h to oxygen-poor static LBE. (b) HAADF STEM image of a FIB foil extracted from position x-x' in Fig. 10a showing the presence of an oxide-filled pocket at the LBE/MAX phase interface. (c) HAADF STEM image of the oxide-filled pocket and accompanying SAED pattern revealing that the oxide is in the vitreous (amorphous) state. (d) STEM EDS elemental maps of the area shown in Fig. 10b showing the presence of thin, seemingly non-continuous oxides (arrows) on the specimen surface.

Fig. 11. (a) BSE image of the Maxthal® 211 ceramic exposed for 3500 h to oxygen-poor, static LBE. (b) HAADF STEM image of the FIB foil extracted from position x-x' in Fig. 11a. The magnified inset shows details from the oxide scale. (c) STEM EDS elemental maps of the area shown in Fig. 11b: the thickness of the oxide scale (arrows) is non-uniform and its continuity might be locally disrupted.

Fig. 12. Overview and profilometry data of the front specimen surfaces prior to and after exposure for 1000 h at 500°C in fast-flowing LBE. The specimens were cleaned from the LBE residues first by hot oil (190°C) and then by a 1:1:1 solution of H₂O₂:C₂H₅OH:CH₃COOH. The red lines on the bulk samples after exposure indicate the location of the cross-sections shown in Fig. 13.

Fig. 13. BSE images of materials exposed for 1000 h to fast-flowing LBE: (a) Zr_2AlC , (b) Zr_3AlC_2 , (c) NZ413 with milled surface finish, (d) 316L SA steel – overview, (e) NZ413 with ground surface finish – overview, (f) 316L SA steel – detail, and (g) NZ413 with ground surface finish – detail. Erosion damages are visible in the 316L SA stainless steel, as opposed to the unaffected NZ413 MAX phase.