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Influence of Corrosion on the Reliability of SMA Materials in the Marine Environment

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Abstract: The research of new materials on the basis of memory shape today occupies the special attention of researchers in various industrial fields, such as medicine, traffic, robotics, etc. This paper analyzes the possibility of applying SMAs (shape memory alloys) in the maritime industry in terms of monitoring the behavior of SMAs in different marine environments. The subject of the research is Cu-Al-Ni and Ni-Ti CC alloys processed by CC (continuous casting) in the shape of bars and Ni-Ti as cast processed by casting in the shape of disk. Using the method of EDX (energy dispersion X-ray spectrophotometry), the chemical composition of alloy elements in zones such as in the air, on the surface of the sea and in the sea was determined after six and twelve months of exposure. By applying the theory of reliability, an assessment of the reliability of the alloy elements was obtained. According to the obtained results of the Cu-Al-Ni alloy, nickel is the most reliable in the sea. Aluminum is the least reliable in the sea. The Ni-Ti CC alloy in the marine environment is more reliable than the Ni-Ti as cast alloy. Based on the changes in the chemical composition of alloys in all three considered zones, it is concluded that all three alloys have the lowest reliability in the sea and the highest in the air.

Key words: SMA, Ni-Ti, Cu-Al-Ni, reliability, marine environment.

1. Introduction

SMAs (shape memory alloys) have been extensively studied due to their advanced properties. With the development of SMA technology such as binary and ternary systems based on Cu (Cu-Zn, Cu-Al, Cu-Zn-Al, Cu-Al-Ni, Cu-Al-Mn), Ni-Ti and high-temperature SMAs usually are used in the automotive, aerospace and medical industries [1]. SMAs use martensitic austenite transformation to achieve superelasticity or pseudoelasticity, OWME (one-way memory effect) and TWME (two-way memory effect) [2]. SMA is characterized by the temperatures of austenite and martensite at the beginning and at the end (As: austenite start, Af: austenite finish, Ms: martensite start, Mf: martensite finish). These characteristic temperatures can be changed by heat treatment or stress [1]. Production technologies of SMA are induction melting, electric arc melting, melt spinning technique, powder metallurgy, combustion synthesis, etc. Products can be hot-worked (forging, rolling), cold-worked (wire drawing, rolling), etc. [3, 4]. In recent years, the CC (continuous casting) technique is one of the production technologies of SMAs based on Cu-Al-Ni thanks to the special competitive mechanism of crystal growth and the creation of a cast product with a favorable texture [5]. Because molten Ni-Ti is highly reactive, it is usually produced by VIM (vacuum induction melting) or VAR (vacuum arc melting). Corrosion resistance can be significantly improved with appropriate surface treatments. In the production of wires, rods, tubes and thin sheets, the alternative processes described above cannot be applied. Such products are still produced by casting, rolling and drawing (wire). If production starts with thick ingots, numerous stages of rolling and intermediate annealing are required to obtain the desired semi-finished product, which is time-consuming and expensive. In such a case, CC may be a preferable

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alternative to ingot casting [6].

Corrosion is defined as the gradual degradation of metal properties caused by a chemical reaction or an electrochemical reaction with the surrounding atmosphere. The physical breakdown of metals is classified as damage, wear and erosion. Metals decompose in contact with moisture/water (H_2O), bases (NaOH, $CaCO_3$, $NaHCO_3$, etc.), acids (HCl, H_2SO_4, HNO_3, etc.), salts (NaCl), liquid chemicals, aggressive metal varnishes and gases (formaldehyde, gases containing sulfur and ammonia) [7]. Seawater is a complex mixture of inorganic salts (mainly NaCl), dissolved gases (especially oxygen), suspended solids, organic matter and organisms [8]. Velocity corrosion in seawater depends on several factors: salt concentration, seawater flow, biofilm on the metal surface in the sea, dissolved oxygen concentration and temperature [5]. Performance is usually discussed depending on the specific zone of the environment, atmosphere, waves, tides, ocean depth and mud [9].

SMA Cu-Al-Ni alloys are susceptible to pitting corrosion, which can occur under deposits and at elevated temperatures [10]. SMA alloys Cu-Al-Ni show susceptibility to the environment and with it the occurrence of corrosion processes such as SCC (stress corrosion cracking), CF (corrosion fatigue) and HE (hydrogen embrittlement) [11]. At water velocities above 2-5 m/s, depending on the composition of the alloy and the layout of the plant, an erosion attack can occur [10]. According to the tests of various authors, it was found that SMA Ni-Ti alloys have good resistance to SCC and good resistance in the marine environment [12]. More recent tests of the Ni-Ti alloy may show a certain susceptibility to pitting corrosion and SCC in calm seawater. Their resistance to erosion corrosion is much better than that of copper-based alloys.

This work includes four chapters. The theoretical part describes the properties and production of Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC memory alloys, as well as their corrosion resistance in the marine environment. The second part describes the material. The third part describes research methods in different settings over a time period of up to twelve months. In the fourth part, the results and discussion are analyzed, i.e. calculations of reliability, failure density and failure intensity are presented based on the systematization of the database of reduced percentages of the original chemical composition of individual alloy elements. At the end, conclusions were performed.

2. Material

Cu-Al-Ni and Ni-Ti alloys are produced by different production techniques. In addition to the usual casting process (as cast) to overcome the creation of a coarse microstructural composition, new processing techniques are applied, such as CC. The samples were made at the Faculty of Mechanical Engineering in Maribor. The experimental device consists of a vacuum induction melting furnace and a vertical continuous wheel [13]. Ni-Ti alloys processed by casting—as cast and CC as well as Cu-Al-Ni alloy processed by CC in Figs. 1a-1c were investigated. The samples were used to investigate the behavior of alloys in different environments—zones in the air, on the surface of the sea and in the sea in the time period after six and twelve months. Eighteen (18) samples were used (three samples for each alloys that were investigated in the period from the beginning of the experiment to six months and three samples for each alloy that were investigated in the period from the beginning of the experiment to twelve months). Fig. 1 shows the alloys from which the investigated samples were cut.

The chemical composition of the elements of Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC alloys was determined by optical inspection of the microstructure using ICP (inductively coupled plasma) analysis and XRF (X-ray fluorescence) analysis [13]. The compositions of the CuAlNi, Ni-Ti as cast and Ni-Ti CC samples were obtained as shown in Table 1 before the start of testing the alloys in the three zones.

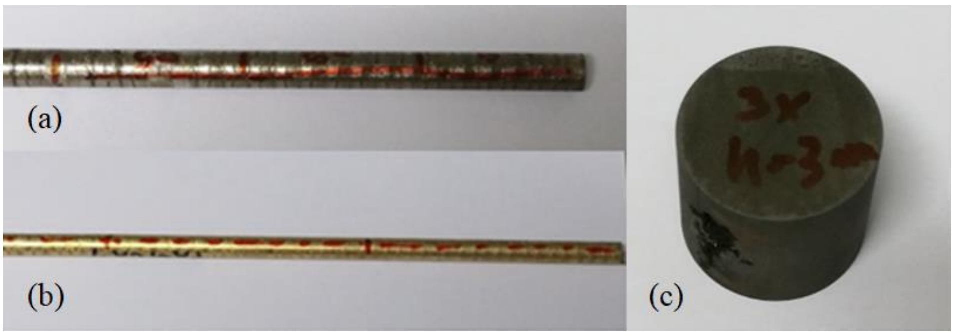

Fig. 1 (a) Ni-Ti CC, (b) Cu-Al-Ni, and (c) Ni-Ti as cast [13].

Table 1 The average value of the elements at the Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC alloys before the start of the test.

Total average value	Elements	Cu	Al	Ni	Ti
Alloys	Cu-Al-Ni	83.89	12.07	4.04	
	Ni-Ti as cast			47.31	52.69
	Ni-Ti CC			53.60	43.53

3. Research Methodology and Methods

The EDX (Energy-dispersive X-ray spectrophotometry) method was used to determine the chemical composition of alloy elements. Research with this intention was carried out at three different locations: 3 m above the water line—i.e. air, in the tidal area, on the surface of the sea and 3 m below the water line—in the sea. Fig. 2 shows the schematic of the conceptual research model of alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC.

The production of samples is presented in the second chapter of the work. After production, the samples shown in Fig. 2 were placed in three different areas. The first area is located 3 m above the water line and represents the area of atmospheric influences. The second area is at the water line and is the area where the tides change. The third area is located 3 m below the water line and represents the marine area. After six and twelve months of exposure under the mentioned conditions, the samples were analyzed by EDX analysis. Then the given calculations were calculated using the reliability theory. The results obtained from these analyses are presented in the next chapter.

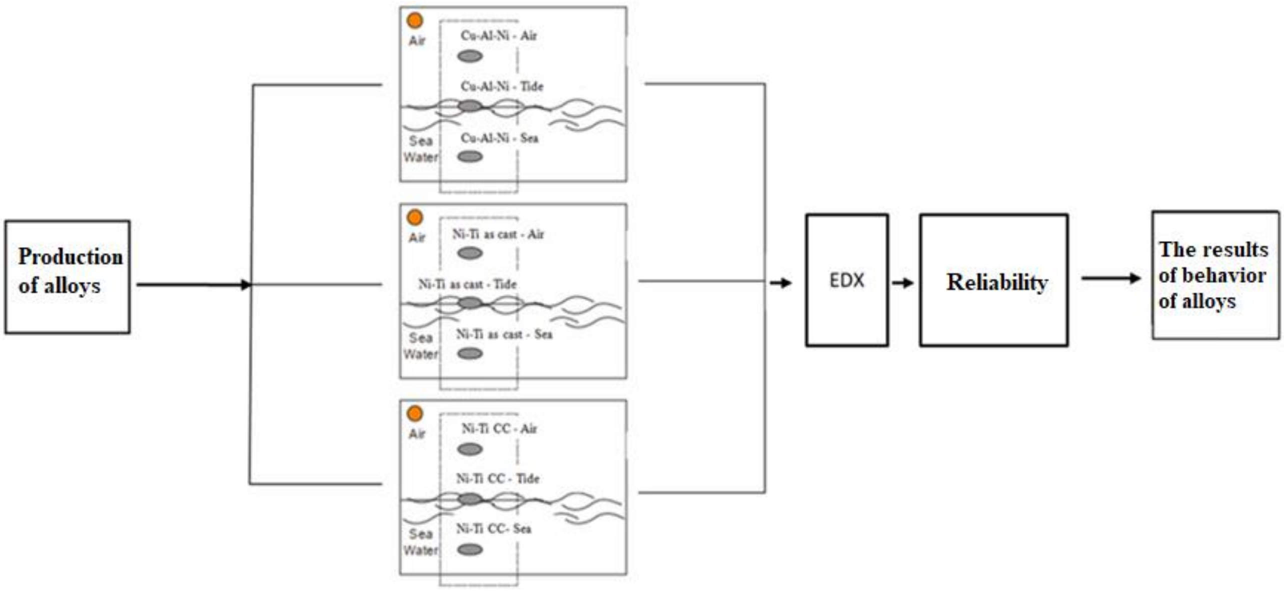

Fig. 2 Scheme of the conceptual research model.

3.1 EDX

The microstructure of the alloys was investigated using a SEM (scanning electron microscope). Along with the SEM, the EDX method of chemical composition can be used to analyze the spectrum of X-rays emitted by the sample when a beam of electrons falls on it. EDX analysis was performed on each sample that was examined in different environmental conditions—each sample number was examined and a certain number of spectra were observed for each sample [14]. Table 2 shows the numbers of samples and spectra for alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC investigated in the period from the beginning to six months in zones 3 m above the water line, on the sea surface and in the sea. In this way, data on the chemical composition of the tested alloys were obtained.

Table 2 Sample numbers and spectrums 3 m above the water line, on the surface of the sea and in the sea for the alloys (a) Cu-Al-Ni, (b) Ni-Ti as cast and (c) Ni-Ti CC in the period from the beginning to six months.

3 m above the water line	Increase (μm)	Number of the spectrums	On the surface of the sea	Increase (μm)	Number of the spectrums	In the sea	Increase (μm)	Number of spectrums
(a)								
Cu-Al-Ni[a]								
Sample 1	200	1-6	Sample 1	200	1-6	Sample 1	200	2-6
Sample 2	100	1-6	Sample 2	100	1-6	Sample 2	200	2-6
Sample 3	70	1-6	Sample 3	70	1-7	Sample 3	100	2-6
(b)								
Ni-Ti as cast[a]								
Sample 1	200	1-6	Sample 1	200	1-5	Sample 1	200	2-7
Sample 2	200	1-6	Sample 2	100	1-7	Sample 1	100	2-7
Sample 2	100	1-6	Sample 3	70	1-5	Sample 2	100	2-7
Sample 3	70	1-6				Sample 2	70	2-7
(c)								
Ni-Ti CC[a]								
Sample 1	200	1-6	Sample 1	200	1-6	Sample 1	200	2-6
Sample 1	70	1-6	Sample 2	100	1-6	Sample 2	200	2-5
Sample 3	70	1-6	Sample 3	100	1-6	Sample 2	100	2-6
						Sample 3	100	2-6
						Sample 3	70	2-8
						Sample 4	70	2-8

[a] Alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC investigated in the period from the beginning to six months.

3.2 Theory of Reliability

Statistical analysis of empirical data is necessary for correct interpretation and prediction of results. There are many statistical methods such as ANOVA (analysis of variance), regression analysis and correlation for data analysis and re-presentation of results. Recent reports are available on the use of statistical methods for cyclic fatigue failure analysis of materials and lifetime prediction. The chemical composition of the alloy elements was determined by the EDX method in the zones in the air, on the sea surface and in the sea after six and twelve months of exposure. Based on the database obtained by EDX analysis, the reliability theory formulations are applied, which recalculates the reliability indicating which alloys are more reliable and which are less reliable, which alloy elements are the most reliable and in which environment. The obtained data were systematized in the Microsoft Excel program, in which all samples and cancellations were collected. Re (reliability), fe (failure density), λ_e (failure intensity) and diagrams were drawn by applying the reliability theory formulations. By applying the reliability theory, an assessment of the reliability of the alloy elements that corrosion will not occur was obtained. Confidence levels were set where a percentage of 50% or 75% was introduced as a measure of the limit of acceptance in which there is a probability that corrosion of the alloys will not occur. The percentage of 75% in the time period from the beginning to six months in different environments (in air, on the sea surface and in the sea) shows that the tested alloys/alloying elements indicate a 75% probability that the corrosion of the alloys will not occur as well as the testing of the alloying elements in period of time from the beginning to twelve months indicates a 75% probability that no corrosion will occur. The percentage of 50% in the time period from the beginning to six months in different environments (in air, on the sea surface and in the sea) shows that the tested alloys/alloying elements indicate a 50% probability that the corrosion of the alloys will not occur as well as the testing of the alloying elements in the time period from the beginning to twelve months indicates a 50% probability that no alloy corrosion will occur.

4. Results and Discussion

As shown in the diagram of the conceptual model in Fig. 2, below will be presented the results of EDX analysis and reliability assessment using the reliability theory obtained for the studied Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC memory alloys. In order to clarify the results presented below, the levels of 50% and 75% reliability, failure density and failure intensity are shown.

4.1 75% Reliability of Copper, Aluminum, Nickel and Titanium at the Cu-Al-Ni, Ni-Ti as Cast and Ni-Ti CC Alloys on the Surface of the Sea and in the Sea

Fig. 3 shows the 75% reliability of copper, aluminum, nickel and titanium of the alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC. Test results after six months indicate the probability that no corrosion will occur. The most reliable alloy is Ni-Ti CC which indicates the lowest achieved values of nickel and titanium in all environments, especially in the marine environment where it indicates the reliability of titanium 27.2% and nickel 25%, and thus the best behavior of the alloy. Low values mean approaching zero values as well as zero itself, which confirms the hypothesis that all diagrams indicate the probability that alloy corrosion will not occur (chapter 3, reliability theory). The Cu-Al-Ni alloy is another reliable alloy, indicating a high corrosion probability (low reliability) of aluminum and copper in all environments, except for nickel which indicates a 20% reliability at the sea surface and 33.3% reliability in the sea. The alloy Ni-Ti as cast is unreliable. In the air, the Ni-Ti as cast alloy indicates a high probability that corrosion will occur, i.e. 89.4% nickel and 88.8% titanium, at the surface of the sea indicates a probability of 67.7% nickel and 60% titanium and in the sea indicates a probability of 55.3% titanium and 50% nickel that corrosion will occur. Numerical values from 50% to 100% represent the range of confidence in which there is a probability of corrosion occurring, the higher the value, the higher the probability that corrosion of the alloy will occur, thus negating the hypothesis that sets the time and probability that it will not occur for a physical quantity to corrosion (chapter 3, reliability theory). In a period of six to twelve months, the reliability of all elements of alloys, aluminum, copper, nickel and titanium in all environments is limited to zero, which means that corrosion will not occur and thus confirms the hypothesis (chapter 3, reliability theory).

Below is a comparison between the four elements: copper, aluminum, nickel and titanium. The most reliable element is nickel (at the Ni-Ti CC alloy) which indicates a reliability level of 33.3% in air and a reliability level of 25% in the sea, as well as nickel (at the Cu-Al-Ni alloy) which indicates a reliability level of 20% on the surface of the sea and 33.3% in the sea, which confirms the hypothesis that corrosion will not occur. The unreliable element is nickel at the Ni-Ti as cast alloy, which indicates a probability of 89.4% in air, 67.7% on the surface of the sea and 55.3% in the sea for corrosion to occur. Another unreliable element is aluminum,

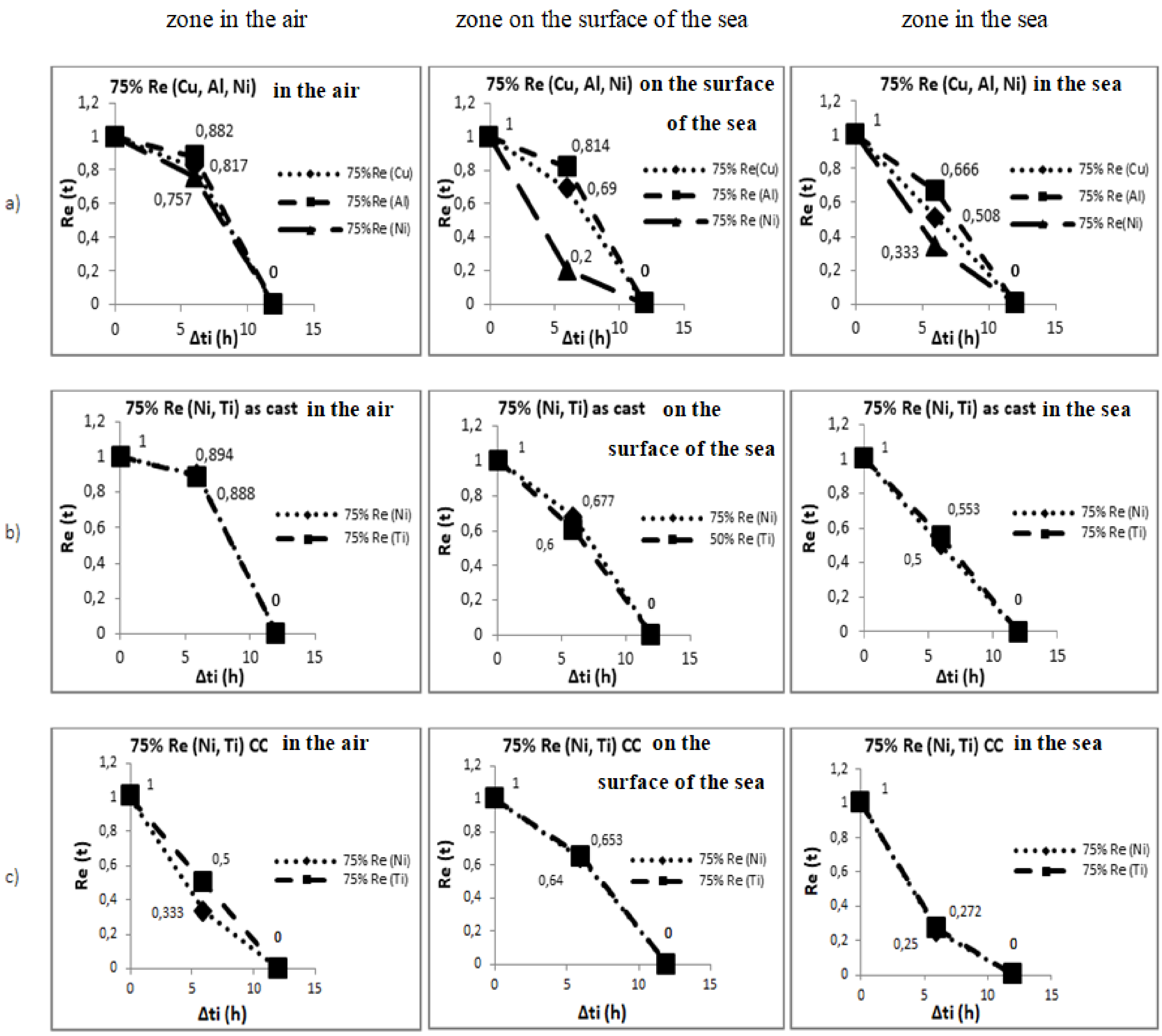

Fig. 3 Reliability (75% Re) of individual elements: copper, aluminum, nickel and titanium at the Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC alloys in different environments (on the surface of the sea and in the sea).

which indicates a probability of 88.2% in air, 81.4% on the surface of the sea and 66.6% in the sea for corrosion to occur. The reason is exposure of the alloy for a long period of time to the corrosive attacks of sea water and thus the wear of the basic and alloyed elements of the alloy (reduction of the percentage of the original chemical composition). The least reliable is the marine environment for all three alloys. The most reliable environment is in air for Cu-Al-Ni and Ni-Ti as cast alloys, and Ni-Ti CC alloys are most reliable on the sea surface.

4.2 50% Reliability of Copper, Aluminum, Nickel and Titanium at the Cu-Al-Ni, Ni-Ti as Cast and Ni-Ti CC Alloys on the Surface of the Sea and in the Sea

Fig. 4 shows the 50% reliability of copper, aluminum, nickel and titanium of the alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC. Test results after six months indicate the probability that no corrosion will occur. The most reliable alloy is Ni-Ti CC, which indicates the lowest values of nickel and titanium in all environments, especially in air where it has a reliability of 28.5% titanium and in a marine environment where it indicates

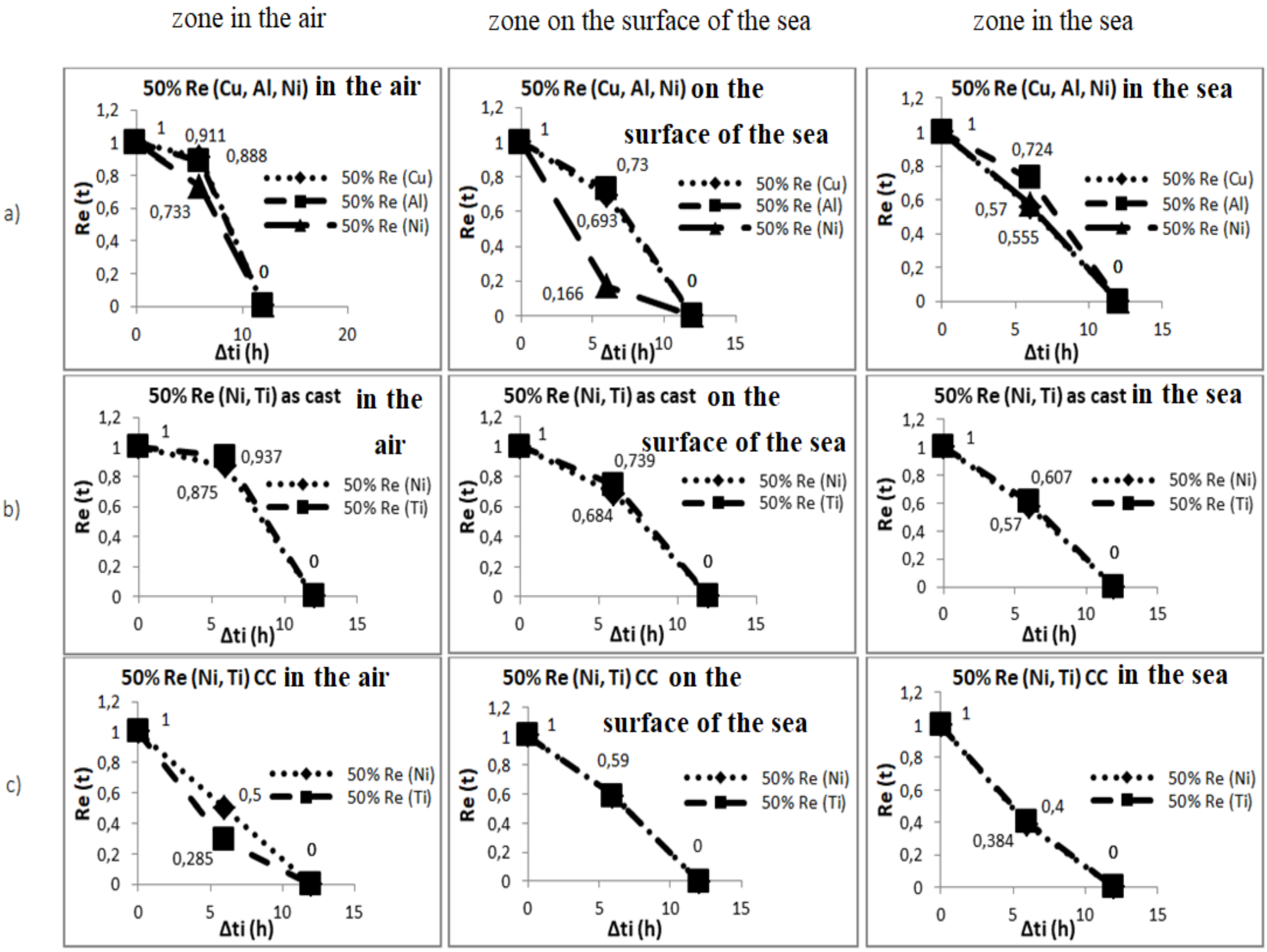

Fig. 4 Reliability (50% Re) of individual elements: copper, aluminum, nickel and titanium at the Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC alloys in different environments (on the surface of the sea and in the sea).

a reliability of nickel 38.4% and titanium 40%. These low values confirm the hypothesis that all diagrams indicate the probability that corrosion of alloys will not occur (Chapter 3, reliability theory). The Cu-Al-Ni alloy is the other reliable alloy, indicating a high probability of aluminum and copper corrosion in all environments, except for nickel which indicates a 16.6% reliability at the sea surface. The alloy Ni-Ti as cast is unreliable. In the air, the Ni-Ti as cast alloy indicates the probability that corrosion will occur, i.e. 93.7% titanium and 87.5% nickel, on the surface of the sea indicates a probability of 73.9% titanium and 68.4% nickel and in the sea indicates a probability of 60.7% titanium and 57% nickel. The higher the values of the obtained results, the higher the probability that corrosion of the alloys will occur, thus negating the set hypothesis that sets the time and probability that corrosion will not occur for the physical quantity (Chapter 3, reliability theory). In a period of six to twelve months, the reliability of all elements of alloys, aluminum, copper, nickel and titanium in all environments is limited to zero, which means that corrosion will not occur and thus confirms the hypothesis (chapter 3, reliability theory).

Below is a comparison between the four elements: copper, aluminum, nickel and titanium. The most reliable element is titanium (at the Ni-Ti CC alloy) which indicates a reliability level of 28.5% in air and nickel with 38.4% in the sea, as well as nickel (at the Cu-Al-Ni alloy) which indicates a reliability level of 16. 6% on the sea surface, which confirms the hypothesis that corrosion will not occur. The unreliable element is titanium in the Ni-Ti as cast alloy, which indicates a probability of 93.7% in air, 73.9% on the surface of the sea and 60.7% in the sea to corrode. Another unreliable element is aluminum, which indicates a probability of 91.1% in air, 73% on the surface of the sea and 72.4% in the sea to occur corrosion. The reason is exposure of the alloy for a long period of time to the corrosive attacks of sea water and thus the wear of the basic and alloyed elements of the alloy (reduction of the percentage of the original chemical composition). The most reliable environment for Cu-Al-Ni and Ni-Ti as cast alloys is in air, and Ni-Ti CC alloys are most reliable on the sea surface. For the Cu-Al-Ni alloy, the least reliable environment is on the sea surface, for the Ni-Ti as cast alloy in the marine environment, and for the Ni-Ti CC alloy in the air and in the sea.

4.3 50% fe (Failure Density) of Copper, Aluminum, Nickel and Titanium at the Cu-Al-Ni, Ni-Ti as Cast and Ni-Ti CC Alloys on the Surface of the Sea

Here, specially selected data from the environment on the sea surface are presented, because they indicated the greatest changes in the chemical composition of the EDX analysis after calculations according to the formulations of the dependence theory. Fig. 5 shows the 50% failure density of copper, aluminum, nickel and titanium of the alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC. The lowest failure density is indicated by the alloy Ni-Ti as cast with a titanium failure density of 4.3% and a nickel failure density of 5.2%. Cu-Al-Ni is the second lowest failure density alloy indicating a low failure density of aluminum and copper, except for nickel which indicates a 13.8% failure density. Ni-Ti CC indicates the highest failure density indicating a uniform chemical composition of nickel and titanium 6.8%. Nickel indicates the highest failure density of 13.8% in the Cu-Al-Ni alloy. Aluminum indicates the lowest failure density of 4.4% and nickel for the Ni-Ti as cast alloy, which indicates a failure density of 4.3%. In a period of six to twelve months, all four elements copper, aluminum, nickel and titanium indicate maximum failure density.

zone on the surface of the sea

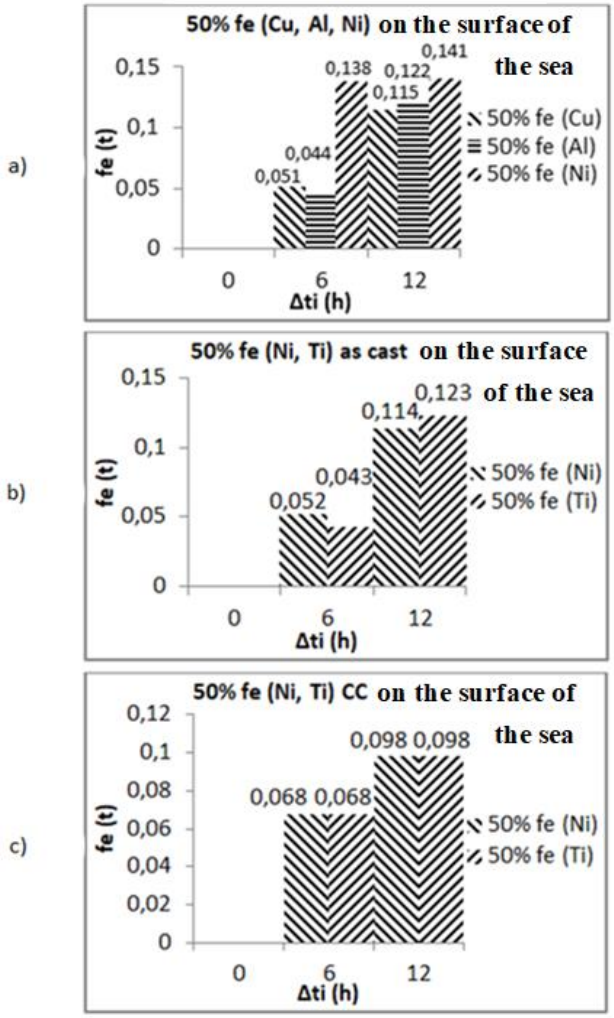

Fig. 5 fe (failure density) of copper, aluminum, nickel and titanium on the surface of the sea.

4.4 75% λ_e (Failure Intensity) of Copper, Aluminum, Nickel and Titanium at the Cu-Al-Ni, Ni-Ti as Cast and Ni-Ti CC Alloys in the Sea

Fig. 6 shows the 50% failure intensity of copper, aluminum, nickel and titanium of the alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC in the sea. The lowest failure intensity is indicated by the Ni-Ti as cast alloy with a titanium failure intensity of 7.4% and a nickel failure intensity of 8.3%. Cu-Al-Ni is the second alloy with the lowest failure intensity indicating a low failure intensity of aluminum 5.5% and copper 8.1%, except for nickel indicating a failure intensity of 11.1%. Ni-Ti CC indicates the highest failure intensity of nickel 12.5% and titanium zone in the sea

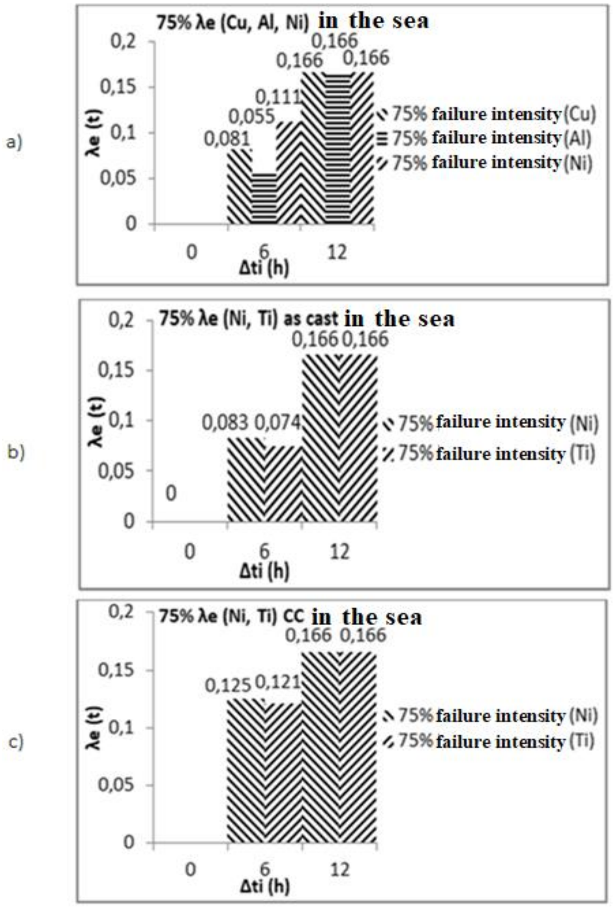

Fig. 6 λ_e (failure intensity) of copper, aluminum, nickel and titanium in the sea.

12.1%. Nickel indicates the highest failure intensity of 11.1% for the Cu-Al-Ni alloy. Aluminum indicates the lowest failure intensity of 5.5% and titanium in the Ni-Ti as cast alloy, which indicates a failure intensity of 7.4%. In a period of six to twelve months, all four elements copper, aluminum, nickel and titanium indicate a maximum failure rate of 16.6% due to exposure to the marine environment for a longer period of time.

5. Conclusion

This paper presents the basic characteristics and the possibility of applying SMA alloys in the marine industry in terms of the reliability assessment of SMAs in different environments with a special focus on the 50% and 75% reliability levels. Two processes for the production of Ni-Ti alloys, disc casting and continuous rod casting, as well as the production of Cu-Al-Ni alloys by CC, are presented. In order to determine changes in the chemical composition of alloy elements, the EDX method was performed after six and twelve months of exposure to the effects of three different environments. The most reliable alloy is Ni-Ti CC in all environments, and the least reliable alloy is Ni-Ti as cast in air and on the sea surface. The Ni-Ti CC alloy in the marine environment is more reliable than the Ni-Ti as cast alloys because nickel and titanium indicate the lowest values, a uniform chemical composition and thus a better behavior of the alloy. Nickel is a more reliable element (at the Ni-Ti CC alloy) at a reliability level of 75% in air, on the sea surface and in the sea. Nickel is the most reliable element (at the Cu-Al-Ni alloy) at a reliability level of 75% in all environments. Nickel is a more reliable element (at the Ni-Ti as cast alloy) at a reliability level of 75% in the sea. Nickel is a less reliable element (at the Ni-Ti CC alloy) at a reliability level of 50% in air and at sea. Aluminum is the least reliable element (at the Cu-Al-Ni alloy) at the 50% reliability level in all environments. Titanium is a less reliable element at the 50% reliability level (at the Ni-Ti as cast alloy) in air and on the sea surface. It can be concluded that the lowest reliability is in the sea, while the highest reliability is in the air. In the future continuation of these researches, it would be good to consider the possibility of analyzing alloys Cu-Al-Ni, Ni-Ti as cast and Ni-Ti CC for a longer period of time, as well as to expand applications in naval facilities.

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