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Obtaining colored oxide layers on titanium alloys with application to utility models

KEYWORDS
Titanium alloys, oxidation, oxide layer, SEM analysis, energy dispersive spectroscopy (EDS) technique.

ABSTRACT
Titanium and its alloys, due to their unusual properties, are widely used in many fields. This paper challenged the design of an everyday use object made of titanium alloy coated with a colored oxide layer. As the object under development, the so-called Italian moka pot was chosen, which is an interesting object due to its history and design.

1. Introduction

Due to its properties, titanium and its alloys are widely used in many areas of life, such as the automotive, shipbuilding, aerospace, chemical, electronics and medical industries. It is worth noting that titanium is an environmentally friendly material, mainly in the aspect of recovery and processing of raw materials. It is a durable material and less subject to wear and tear during daily use. Compared to other materials, extracting titanium from the earth's crust uses less energy, and it is also a recyclable material. It also finds its use in the production of everyday items and in construction and architecture [1–3]. Leading examples include buildings such as the Guggenheim Museum in Bilbao (Figure 1.) built from titanium, glass and limestone, the Science Mall, i.e. the largest of the Science Center buildings in Glasgow (Figure 2), which is covered with titanium, and the Koetsu-Ji temple in Kyoto where a titanium sheet imitating the appearance of burnt tiles was used as the roofing (Figure 3) [4–6]. Among the surface layers that enhance the aesthetic features (including anti-corrosion properties) of titanium alloy parts, the most widely used are colored oxide layers (Figure 4) [7].

Figure 1. Guggenheim Museum in Bilbao

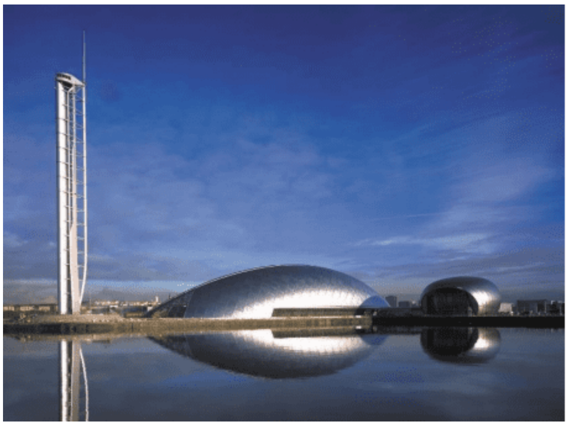


Figure 2. Science Mall of the Glasgow Science Centre

Figure 3. Koetsu-Ji Temple in Kyoto with titanium sheeting imitating burnt tile

Figure 4. Dog Elongate, HUNG Yi

Using various methods of modifying the surface layer of titanium and its alloys, a variety of colors can be given to the parts (Figure 5). The factor influencing the resulting color is the thickness of the resulting oxide layer. The oxide layer itself is transparent and the visible color is the result of interference of rays reflected from the oxidized surface [8]. To obtain colored layers, the most common methods are anodizing or annealing at high temperatures.

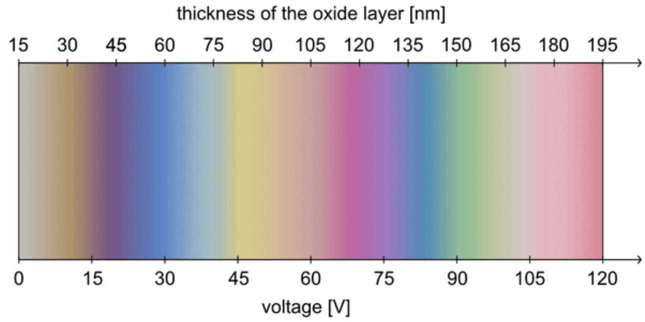


Figure 5. Graph showing the colors obtained depending on the voltage used and the thickness of the oxide layer [8]

2. Experimental studies

Ti-25Al-12.5Nb titanium alloy was used for the study. Chemical composition of the alloy is indicated in Table 1, and the surface of the sample before oxidation is shown in Fig. 6.

Table 1. Chemical composition of the investigated alloy

Elements, % at.				
Al	Nb	Mo	V	Ti
25,0	12,5	6,01	0,48	rest

Figure 6. Surface of the sample before oxidation

In order to obtain colored oxide layers, the prepared samples were oxidized at 700° for 50, 100, 300 and 500 hours, and then cooled in still air. Observations of the surface of the samples and chemical composition analyses were carried out using a TESCAN VEGA 4 scanning electron microscope.

The annealing resulted in a material with varying color. The sample annealed for 50h (Figure7) obtained colors such as green, yellow and pink. Purple shades and dark green can also be seen. In this case, the most saturated colors were obtained. On the surface of the sample annealed for 100h (Figure8), the predominant colors are turquoise and purple. In cold light, a dark blue color can also be observed, while in warm light an orange color appears. The sample oxidized at 300h (Figure 9) obtained the least saturated colors. It is hard to see colors other than shades of brown. In some places the green color gently shines through, with additional yellow in warm light. The last of the samples, oxidized for 500h (Figure 10), has a predominance of turquoise and blue colors. To a lesser extent, light green color is also present on the surface, as well as brown. Under warm light, purple can also be seen on the sample.

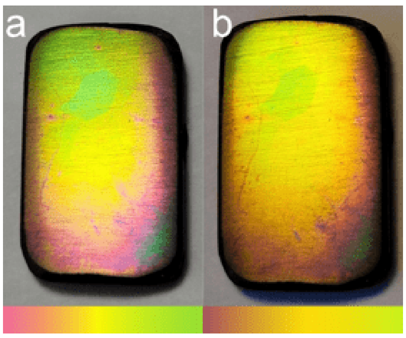


Figure 7. Surface of the oxidized sample during 50 h; a) in cold light, b) in warm light

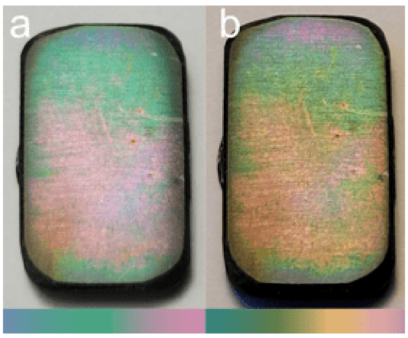


Figure 8. Surface of the oxidized sample during 100 h; a) in cold light, b) under warm light

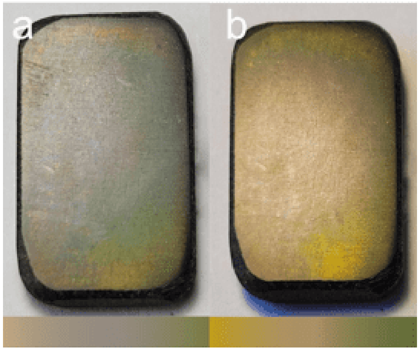


Figure 9. Surface of the oxidized sample during 300 h; a) in cold light, b) in warm light

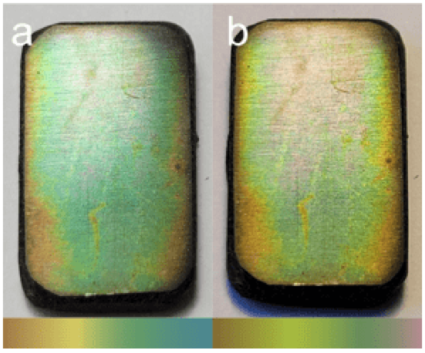


Figure 10. Surface of the oxidized sample during 500 h; a) in cold light, b) in warm light

Observations using a scanning electron microscope made it apparent that an oxide layer with a specific structure is formed on the surface of the samples (Fig. 11), characterized by a rather irregular structure in the form of specific oxide eruptions. The EDS analysis (Figs. 12–15) performed made it possible to identify chemical elements. The dependence of the percentage share of individual elements on the annealing time is shown in Figures 16–17. It is noted that the oxygen content on the surface of the samples increases with the oxidation time. At the oxidation time of 50–100h, a significant increase in oxygen content is observable, while at the time of 300–500h the increase is small. This may mean that beyond about 300h, further oxidation does not result in a large increase in oxygen content, and thus an oxidation time of several hundred hours is limiting for the subsequent effects of the oxide layer. Similar conclusions can be drawn from the titanium content, which significantly decreases in the range of 50–100h of oxidation, and with the oxidation time the decrease is smaller and smaller. The percentage of the other elements, namely carbon, aluminum, vanadium, niobium and molybdenum, is almost constant, and does not change significantly.

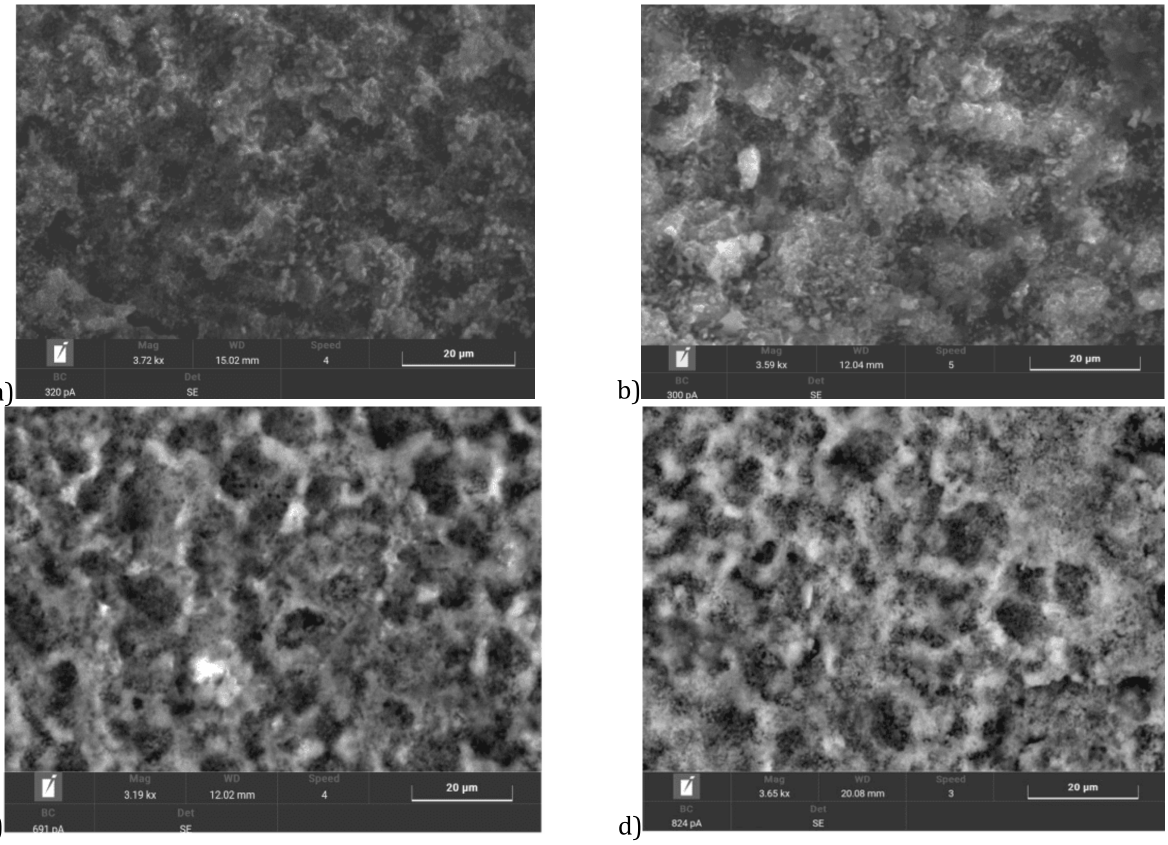
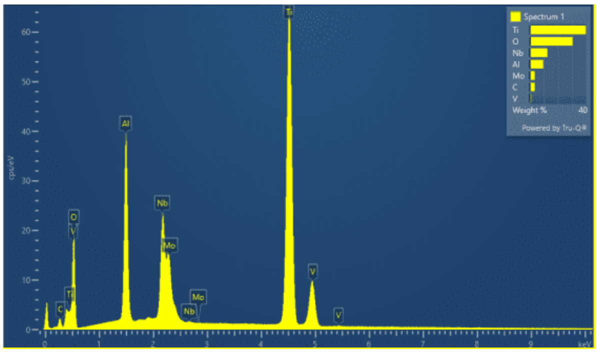
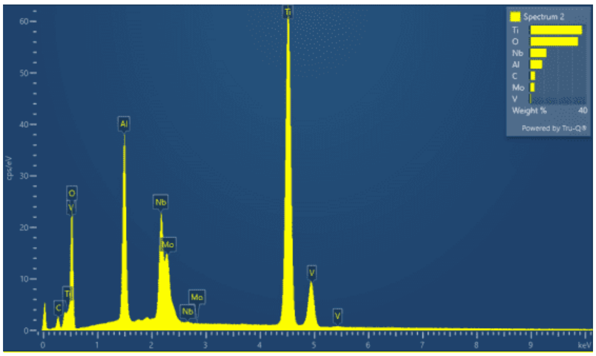


Figure 11. SEM image of the surface for the sample oxidized during (a) 50h, (b) 100h, (c) 300h, (d) 500h



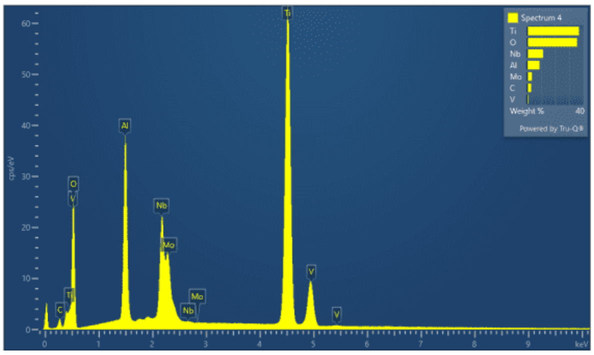
Element	Line Type	Wt%	Atomic %
C	K series	3.43	8.04
O	K series	30.40	53.42
Al	K series	9.50	9.89
Ti	K series	39.81	23.36
V	K series	0.92	0.51
Nb	L series	12.49	3.78
Mo	L series	3.45	1.01
Total:		100.00	100.00

Figure 12. EDS analysis for the sample oxidized during 50h



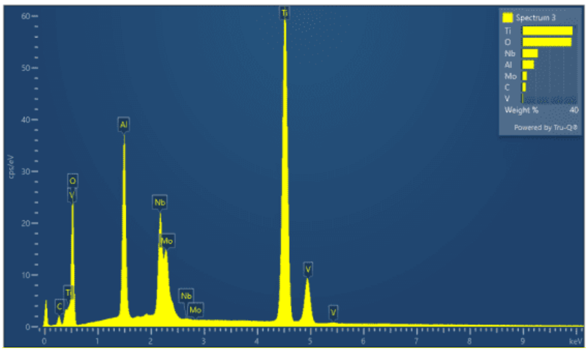
Element	Line Type	Wt%	Atomic %
C	K series	3.78	8.42
O	K series	34.24	57.31
Al	K series	8.74	8.68
Ti	K series	37.13	20.76
V	K series	0.90	0.47
Nb	L series	11.85	3.42
Mo	L series	3.37	0.94
Total:		100.00	100.00

Figure 13. EDS analysis for the sample oxidized during 100h



Element	Line Type	Wt%	Atomic %
C	K series	2.80	6.23
O	K series	35.72	59.69
Al	K series	8.82	8.74
Ti	K series	37.03	20.67
V	K series	0.87	0.45
Nb	L series	11.37	3.27
Mo	L series	3.39	0.95
Total:		100.00	100.00

Figure 14. EDS analysis for the sample oxidized during 300h



Element	Line Type	Wt%	Atomic %
C	K series	2.82	6.28
O	K series	35.86	59.88
Al	K series	8.69	8.61
Ti	K series	36.66	20.45
V	K series	0.92	0.48
Nb	L series	11.50	3.31
Mo	L series	3.54	0.99
Total:		100.00	100.00

Figure 15. EDS analysis for the sample oxidized during 500h

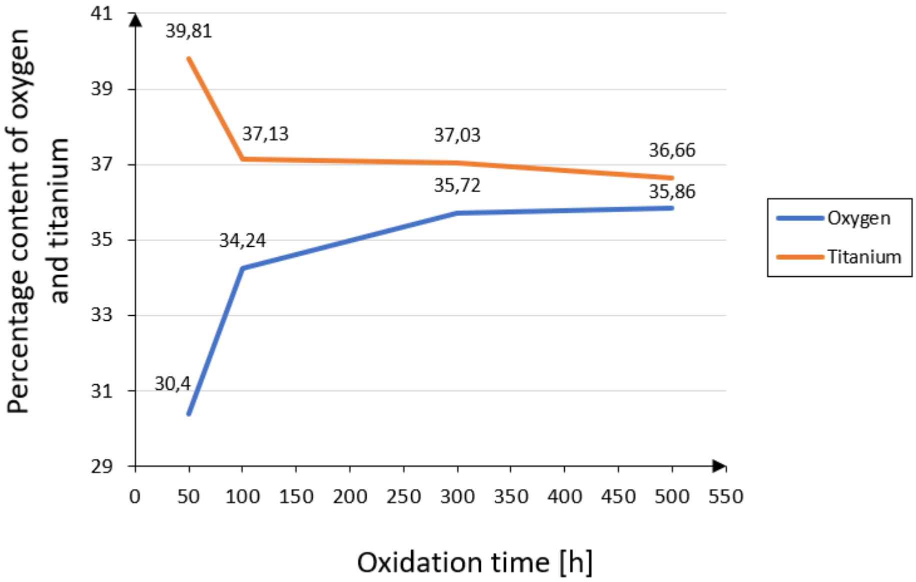


Figure 16. Graph showing the dependence of oxygen and titanium content percentages on oxidation time

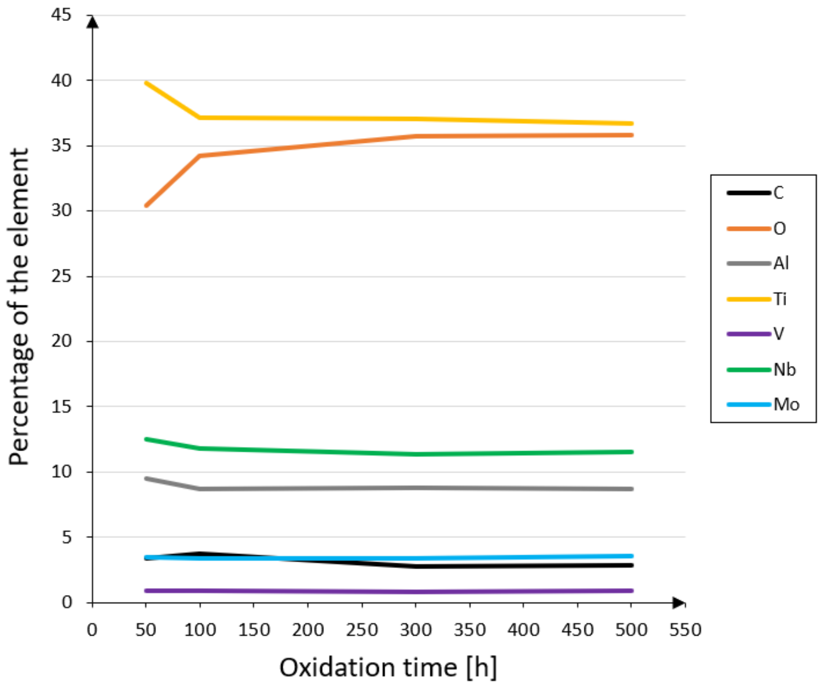


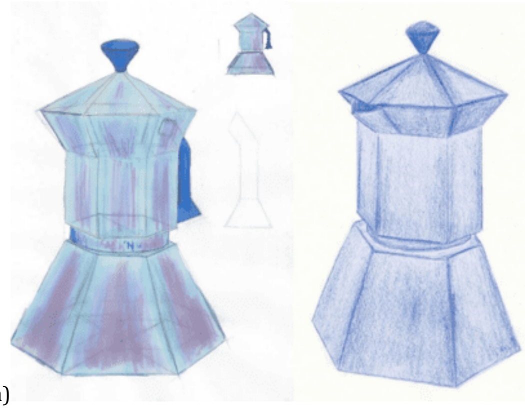
Figure 17. Graph showing the percentage of elements as a function of oxidation time

3. Functional form design

The main design consideration was to make a coffee pot from titanium alloy coated with a colored oxide layer to enhance the aesthetic value of the design. Another assumption was to make the equipment in a way promoting ergonomic work, that is, suitable for preparing coffee on gas and electric hobs, with a capacity equivalent to four cups of coffee.

3.1. Conceptual work

The first design concept (Fig. 18a) resembles in form the original Bialetti moka pot. This coffee pot was the main inspiration in creating the design. The body of both vessels has the base of a regular hexagon, the lower vessel is a pyramid, while the upper vessel adopts the shape of a pillar with a prism-shaped stem with a pyramid-shaped head. The lid also adopts the shape of a pyramid, and the handle has geometric shapes that relate to the form of the entire coffee pot.


a)

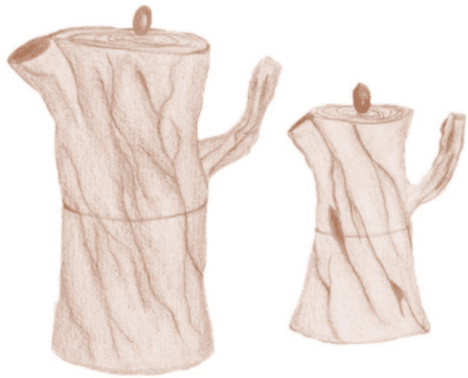

b)

Figure 18. Conceptual sketches

The second concept (Figure 18b) is inspired by nature, the shape of the coffee pot refers to the trunk of a tree or the branches of a coffee bush. The handle and the opening through which the coffee is poured have the form of a branch growing out of the trunk. In turn, the knob for opening the lid has the shape and color of a coffee fruit.

3.2. Creation of a model using CAD techniques, technical documentation and visualizations

The moka pot models were created in Autodesk Inventor Professional 2021. A technical drawing and design visualizations were also prepared using the same software (Fig. 19). The target design selected is the second design (Fig. 20). It was prepared in the form of an assembly model, made with attention to detail, taking into account all part fastenings and the thread that connects the upper vessel to the lower one.

Figure 19. Project visualization

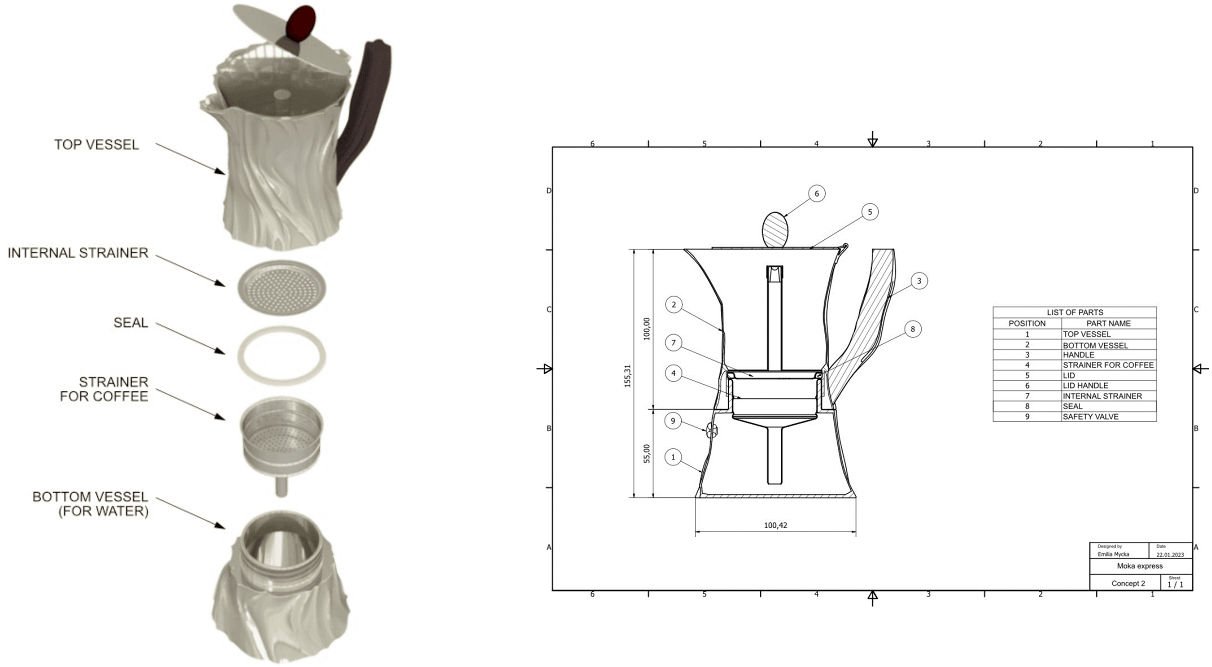


Figure 20. Assembly model of the selected design

4. Summary

Titanium alloys are food-grade materials that are increasingly used for various types of cookware. These materials have high corrosion resistance, and are also good heat insulators, reducing heat loss in the cookware when heating its contents.

As a result of exposure to high temperature on the material, oxide formation occurs during annealing, and the thickness of the oxide layer formed depends on the timeand the temperature used. The growth rate of the oxidation products strongly depends on the temperature, and the diffusion processes taking place activate as the temperature and oxidation time increase.

The designed unit measures 180 mm in height and 130 mm in width. The height of the lower vessel is 55 mm, while the upper vessel is 100 mm. The dimensions chosen mean that the capacity of the moka pot is equivalent to 4 cups of coffee. The material from which the device's vessels would be made is Ti-25Al-12,5Nb-6,01Mo–0,48V alloy. It is also assumed that the alloy would be covered with an oxide layer obtained by oxidation during 50 h at 700 ºC, resulting in green, yellow and orange on the surface of the vessels.

5. Literature

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