## 3.1 Final publishable summary report

### Executive summary

The main objective of the project is to develop a new aluminium wrought alloy that can work at temperatures up to 200-250ºC. The European aeronautical industry envisages that for near future applications the service temperature of some aluminium components that are part of the air conditioning system of the airplanes will increase to up to 250ºC. Both casting and wrought alloys and thermal treatments will have to be developed for those applications. Furthermore and in order to maintain the competitiveness of the European industry a special attention will be required in terms of cost and sustainability of these developments.

In order to carry out this project a consortium formed by three participants was established. Fundación Tecnalia, Fundación Inatec and Refial S.L., all of them based in the Basque Country region in Spain. The coordinator is Fundación Tecnalia.

Two formulations based on Al-Cu alloys were selected at the end of the first year of the project as the most promising alloys to be used at high temperatures. In this second year, the heat treatment parameters were optimized for each of these formulations and they were mechanically and metallurgically characterized. The ageing behaviour of both alloys was also studied, and also the castability, conformability and recyclability of them. Finally, a technical, environmental and cost analysis was performed in order to check the suitability of any or both of the developed alloys to be used in applications requiring long-time stay at high temperatures.

Following the main results achieved in the project are listed:

- Casting of samples with 24 different compositions following the design of experiments approach planned.
- Positive results in the preliminary analysis with tensile properties of extruded specimens at room temperature and 250ºC.
- Selection of the optimum ageing parameters for the heat treatment of the two formulations selected at the end of the first period of the project.
- Results of the formability study of both alloys performed with the use of the Gleeble 3800 thermomechanical analyser.
- Results of the recyclability study concluding that developed alloys have similar recyclability to other Al-Cu wrought alloys.
- Tensile testing results of the extruded samples after applying the optimum heat treatment confirming the increase in strength after the heat treatment due to the precipitation of $Al_2CuMg$ particulates.
- Creep testing results showing better creep behavior at all studied stresses for the high copper content alloy.
- Ageing behavior of the alloys showed that strength is reduced between 45 and 55% depending on the composition due to the coarsening of $Al_2CuMg$ to form a continuous phase in the grain boundaries.
- Tensile results after the 1000-hour ageing process showed that strength of the developed alloys is slightly higher at 250ºC to other alloys except wrought alloy 2219 and casting alloy AU5NKZr.
- The production cost of the high copper content alloy is slightly higher (0.35 €/Kg) than for the low copper content alloy and both of them are more expensive than the reference 2618 alloy (40 and 50% more expensive respectively).

## Introduction. Description of project context and objectives

The main objective of the ALT project is to develop a new aluminium wrought alloy that can work at temperatures of up to 200-250ºC. The European aeronautical industry envisages that for near future applications the service temperature of some aluminium components that are part of the air conditioning system of the airplanes will increase to up to 250ºC. Both casting and wrought alloys and thermal treatments will have to be developed for those applications. Furthermore and in order to maintain the competitiveness of the European industry a special attention will be required in terms of cost and sustainability of these developments.

Several heat resistant aluminium based materials that have been developed in the last years do exist that might already be used in those temperature ranges. However, most of them are either based on the incorporation of expensive alloying elements such as rare earths or the addition of ceramic reinforcements (SiC, $Al_2O_3$, $B_4C$ etc.).

Therefore the development of a cost efficient aluminium alloy for high temperature applications produced by ingot metallurgy presents much industrial and economical interest and potential impact.

The project aimed at developing such alloys through a methodology based on the identification of the effect of up to 12 alloying elements and their combinations in the properties of aluminium alloys and the selection of the optimum combination of these alloying elements through the Taguchi methodology. In a first stage gravity casting samples with 24 different formulations are cast by gravity casting and preliminarily tested in order to check whether any of these combinations may reach the established specifications in terms of tensile properties at high temperatures (200-250ºC) and metallurgical soundness.

Subsequently, during the second stage of the project the most promising 2 alloys have been further developed through additional castings, optimization of thermal treatments and complete characterization and cost analysis. The project has included tasks related to the study of the ageing behaviour, forming maps, fatigue and corrosion resistance of the developed alloys.

### 1st phase : Selection of alloys

First of all, a bibliographical review in the field of high temperature aluminium alloys was carried out. Using the information collected during the bibliographical review and the previous knowledge of the consortium, a list of twelve alloying elements was selected, taking also into account the restriction of cost that the developed alloy must have. In order to prepare the Design of Experiments (DoE) based in the Taguchi methodology, the ranges of the alloying elements was selected.

Subsequently the preliminary castings were made in the foundry pilot plant of Tecnalia in month 7 and the preliminary characterization has started. For each composition of the DoE, the developed alloy has been cast in two different moulds. These moulds were designed in order to obtain parts with cylinder shape. For each composition two small cylinders were cast in a mould with a diameter of 70mm, and a bigger cylinder was also cast in the mould with a diameter of 150mm. One of the small cylinders was left in the “as-cast” state and six specimens were machined for tensile testing, while the other small cylinder was thermally treated before machining the six specimens. The biggest cylinder was extruded obtaining a thinner cylinder with a diameter of 50mm. The extruded part was cut in smaller pieces. From some of these pieces tensile specimens were machined to be tested in the "as-extruded" condition, while some others were thermally treated to be tested in the "extruded+thermally treated" condition.


Figure 1. (a) Melting of pure Al in the induction furnace, (b) molten Al in the SiC crucible.

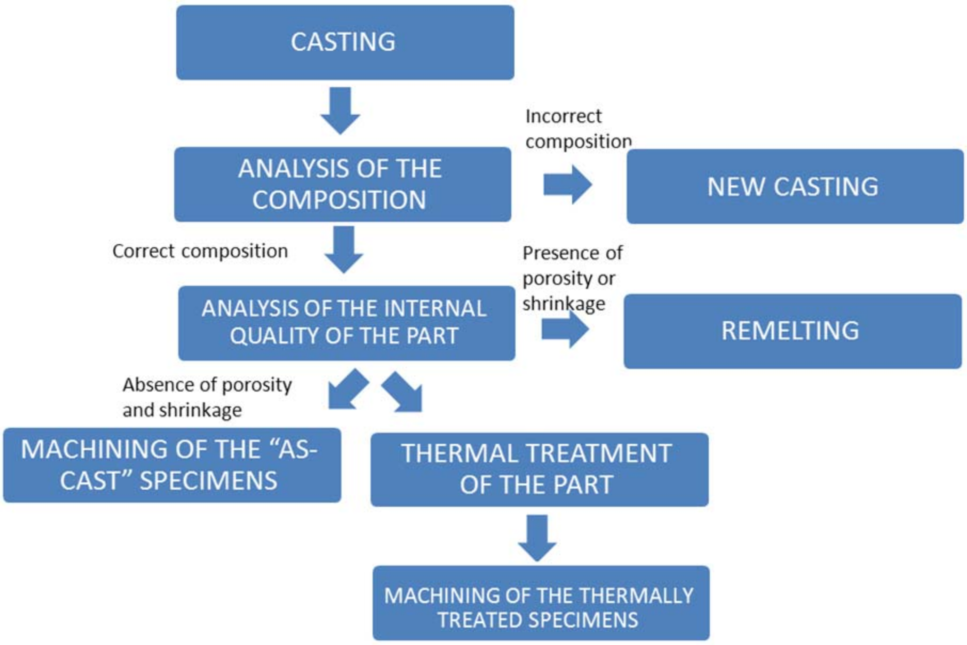


Figure 2. Validation scheme for the castings

24 large cylinders with a diameter of 150mm and a height of 120mm were obtained after the casting task.

The next step was the preliminary characterization. In order to decide the most promising alloys among the 24 different alloys during this first part of the project, a preliminary characterization of all the compositions in different conditions was carried out. This preliminary characterization consisted on: Tensile tests at 25ºC (RT), tensile tests at 250ºC (HT) after an ageing treatment of 100 hours and Microstructural analysis.

The results obtained in the first phase of the project were promising and two alloys were selected to continue with the second stage of the project. Both alloys overcame the evaluation criterion that was established at the beginning of the project and it was expected that further optimization of the thermal treatment and extrusion processes may provide further improved properties.

**2nd phase development and validation of two high temperature resistant aluminium alloys**

To finish the task of developing the alloys formability and recyclability studies of the alloys were performed. The conformability study was carried out in the Gleeble and concluded that the conformability is similar for both alloys, with a similar cracking severity after the compression tests.

Both alloys were subjected to an ageing process of 1000 hours at 250ºC in order to check the evolution in the mechanical properties. For both alloys, an important loss in tensile properties was observed. For the FC1 alloy this reduction was calculated to be a 43% on both yield and tensile strength. This loss of strength resulted in an important increase in the elongation, from 15 to 33%. For FC2 alloy, the loss of strength was even higher, reaching a 55% approximately. The elongation increased from 8,4 to 24%.

Finally, a technical, environmental and cost evaluation was performed. Comparing the tensile properties of the developed alloys after being subjected to an ageing process of 1000 hours with the properties of some commercial alloys used at high temperatures in the same condition, it was concluded that the strength of the two developed alloys is slightly higher at 250ºC than the other alloys except wrought alloy 2219 and casting alloy AU5NKZr that have higher yield and tensile strength.

Tensile properties of the two developed alloys after being subjected to an ageing process of 1000 hours where compared with the properties of commercial wrought alloys obtained from bibliography and with the properties of the casting alloy AU5NKZr, supplied by Liebherr.

The environmental evaluation was focused in the recyclability of the developed alloys as well as the production cycle analysis, concluding that the recyclability of the two alloys developed in the project is comparable to that of other Al-Cu alloys, being lower the recyclability indexes for the higher copper content alloy.

Finally, the production cost of the two alloys was compared. In this cost analysis the cost of personnel, consumables, maintenance, energy and equipment were taken into account, but further processing steps such as filtering, degassing, heat treatment or control were not included in the pricing. Comparing the price of the two alloys, FC2 has a higher cost due to the higher content of copper.

| Alloy | Cost (€/kg) |
|---|---|
| FC1 | 3,65 |
| FC2 | 3,90 |

Table 1: Cost of the raw materials required to produce the selected alloys

Following the main results achieved during the second year of the project are listed:

- Selection of the optimum ageing parameters for the heat treatment of the two formulations selected at the end of the first period of the project.
- Results of the conformability study of both alloys performed in the Gleeble.
- Results of the recyclability study concluding that developed alloys have similar recyclability to other Al-Cu wrought alloys.
- Tensile testing results of the extruded samples after applying the optimum heat treatment confirming the increase in strength after the heat treatment due to the precipitation of $Al_2CuMg$ particulates.
- Creep testing results showing better creep behavior at all studied stresses for the high copper content alloy.
- Ageing behavior of the alloys showed that strength is reduced between 45 and 55% depending on the composition due to the coarsening of Al2CuMg to form a continuous phase in the grain boundaries.
- Tensile results after the 1000-hour ageing process showed that strength of the developed alloys is slightly higher at 250ºC to other alloys except wrought alloy 2219 and casting alloy AU5NKZr.
- The production cost of the high copper content alloy is slightly higher (0.35 €/Kg) than for the low copper content alloy and both of them are more expensive (40 and 50% respectively than 2618 alloy.

## Description of the main S&T results/foregrounds

The technical activity foreseen in the project had been divided in four different workpackages. Following the description of the most relevant work carried out in each of these workpackages is provided together with a detailed summary of the main results.

### WP1: Formulation of high temperature resistant aluminium alloys

The work began with a complete state of the art of aluminium alloys for high temperatures and the analysis of the information provided by the Topic Manager on the requirements of the new alloys.

Using the information collected during the bibliographical review and the previous knowledge of the consortium, a list of twelve alloying elements was selected, taking also into account the restriction of cost that the developed alloy should have. This task was critical as the correct selection of the alloying elements as well as the maximum and minimum amounts was to have a direct influence in the microstructure and performance of the 24 alloys to be produced. The criteria used to carry out the final selection was the study of the common alloying elements in wrought Al alloys, the analysis of the influence of each element and the wish of the Topic Manager of not using expensive elements such as lithium and scandium. These elements are known to have a large positive impact on the high temperature performance of aluminum alloys but the TP wished to work with rather conventional alloying elements and to leave the possibility of applying the former in case the desired level of properties were not reached at the end of the first phase of the project. Eventually and luckily the obtained results with conventional chemical elements were positive and the project has continued with the original plan.

Furthermore in order to prepare the Design of Experiments (DoE) based in the Taguchi methodology, the ranges of the alloying elements was selected. For this selection, the composition of all the commercial alloys of the Al-Cu system (series 2xxx alloys) was studied and the minimum and maximum contents of the selected elements in these alloys were used as reference values for the selection of the ranges for the DoE.

The DoE gives the composition of the 24 alloys that were developed in the task T1.3 of the project.

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Cu | Mg | Fe | Ni | Mn | Si | Ti | Zr | Cr | V | Sn | Zn |

Table 2: Selected alloying elements.

The preliminary castings were successfully prepared and samples were sent to be extruded.

For each one of the 24 different alloys of the DoE, tensile tests and metallurgical analysis were carried out in four different conditions in order to analyze the influence of the thermal treatment and the extrusion in the mechanical properties and microstructure of the alloys. These four different conditions are the following.

- “As-cast”
- Thermally treated.
- “As-extruded”

- Extruded+thermally treated.

For the “as-cast” and “thermally-treated” conditions, a cylindrical mould with an internal diameter of 70mm and a height of 250mm was designed. With this mould two cylindrical parts of around 2.5 kgs each were cast for each composition. From one of these cylinders the “as-cast” specimens were machined for the tensile testing (task 1.4), while the other cylinder was subjected to a T6 thermal treatment (subtask 1.3.5) before machining the specimens.

For the “as-extruded” and “extruded+thermally treated” conditions, another cylindrical mould was designed with an internal diameter of 150mm and a height of 120mm. This way it was possible to extrude the samples with an extrusion ratio of around 1:15, obtaining larger cylinders with a diameter of 40mm and a length of around 850mm after removing the two extremes of the cylinder.

### Castings

Starting in November 2012, the 48 castings were prepared.


Figure 3: (a) Melting of pure Al in the induction furnace, (b) molten Al in the SiC crucible.

In order to validate each of the cast cylinder, first of all the composition of each casting was analysed. In case this composition was not the one expected, the casting was repeated again. In case the composition was the one defined in the DoE, the part containing the solidification shrinkage was cut and removed. Once cut, the quality of the sample was visually inspected. In some samples, small pores due to the solidification shrinkage could still be seen, so these cylinders were remelted and cast again at a lower temperature in order to reduce this shrinkage as much as possible. In case the section of the cylinders was free of pores, the sample was either sent to be machined or kept in order to be thermally treated. From each composition, one of the cylinders was machined to obtain 6 tensile specimens in the “as-cast” condition from it and the other one was kept.

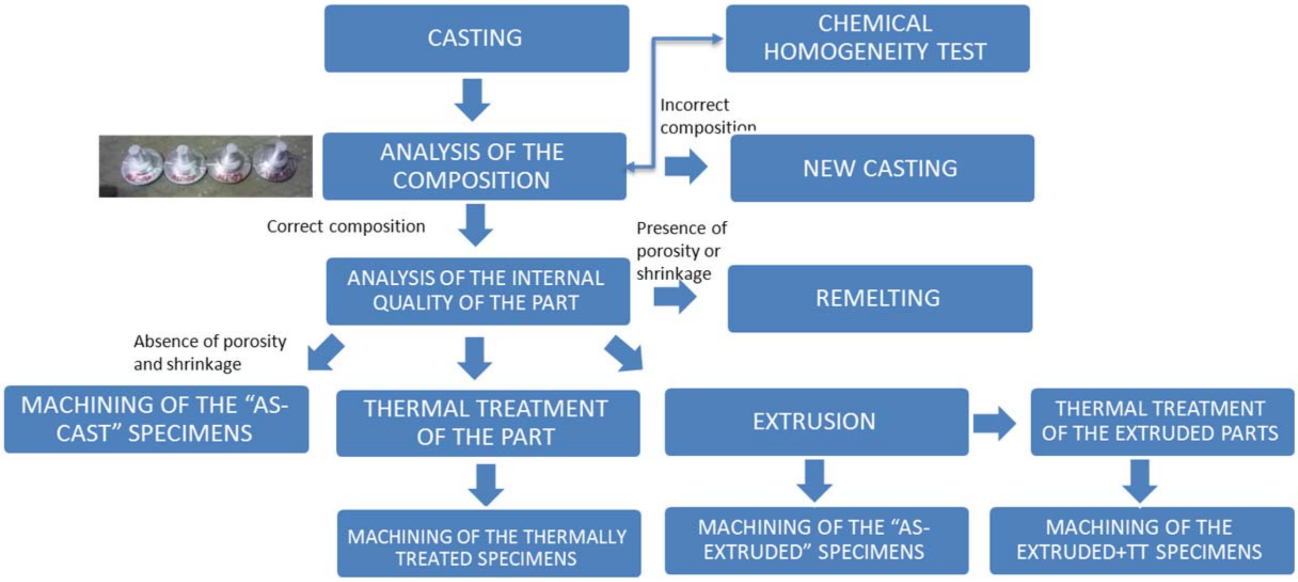


Figure 4: Validation scheme for the castings

Subsequently half of the samples were thermally treated. The consortium, together with the topic manager, decided that a general T6 treatment would be used to treat all the samples. This treatment is the most usual for hardening Al-Cu alloys and it consists in three phases: The heat treated samples were then machined in order to obtain tensile specimens from them to be tensile tested.

Extrusion

24 big cylinders with a diameter of 150mm and a height of 120mm were obtained after the casting task. The top part of the cylinder concentrated the solidification shrinkage so it was removed and the rest of the cylinder (80mm height) was sent to the extrusion process (figures 5 and 6).


Figure 5: Cast big cylinder (a) after demoulding and (b) after removing the top part.


Figure 6: Extruded cylinders in the (a) as-extruded and (b) extruded + heat treated condition.

Subsequently the preliminary characterization took place. Both metallurgical analysis (Scanning electronic microscope and optical microscope analysis) and the tensile tests at room temperature and 250ºC were carried out in order to select two alloys for the next phase of the project.

The final decision on the selection of the two alloys to be used in the second year of the project was completed in month14. The analysis of the results was started with a preliminary selection of various alloys. In order to make this preliminary selection a criterion was established:

1.- High values of yield strength and tensile strength at 250ºC. As the main objective is to develop an aluminium alloy with high elevated temperature strength, the analysis was focused in the values obtained in the tests at high temperatures. The following limits were established:

| | As-cast | HT | As-extruded | Extruded+HT |
|---|---|---|---|---|
| Ys(Mpa) | 100 | 130 | 80 | 125 |
| UTS (Mpa) | 125 | 170 | 100 | 150 |

Table 3. Threshold values of yield strength and UTS for the different conditions.

2.- Low difference between values of yield strength or tensile strength at 250ºC and room temperatures.

3.- Low dispersion in the results.

In month 12 only the results from the tests of samples in the as cast condition were available.

The results in the as-cast condition can be graphically seen in the following figures.

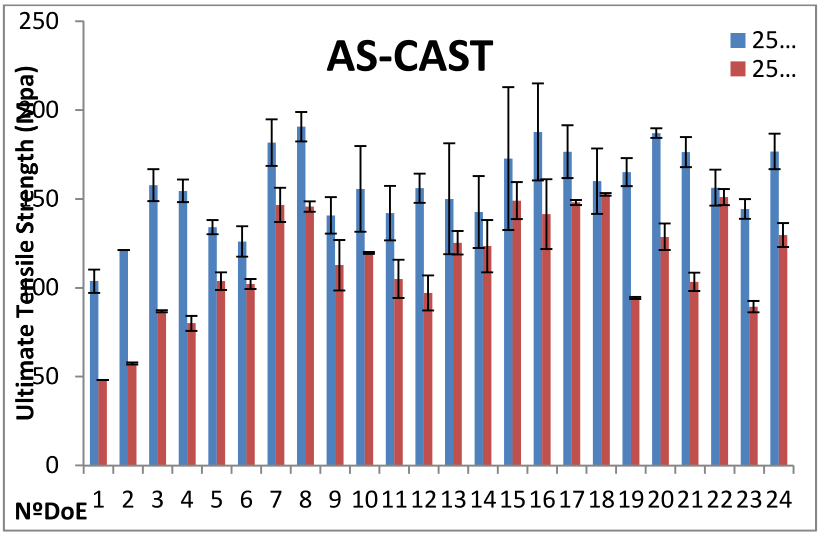


Figure 7: Ultimate tensile strength values in the as-cast condition.

Analysing these results, it could be preliminarily concluded that the compositions that fulfil the criteria established for the preliminary selection of the alloys are the following: 7, 8, 13, 15, 16 and 20.

The results in the heat treated condition can be seen in the following figures:

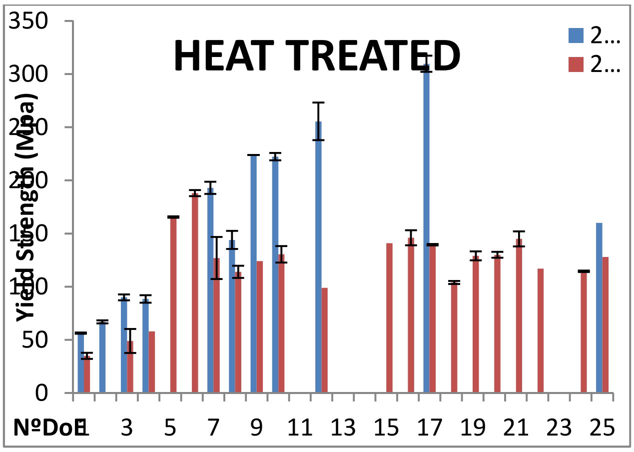


Figure 8. Yield strength values in the heat treated condition.

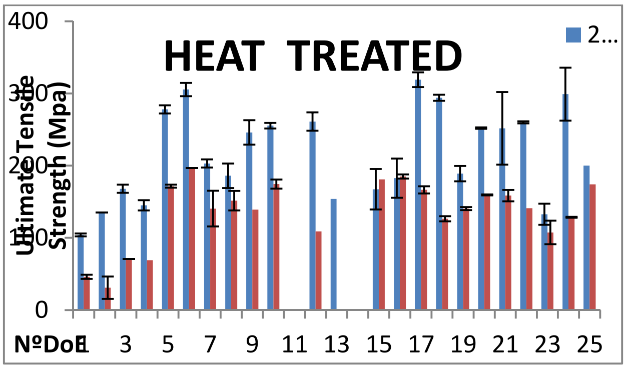


Figure 9. Ultimate tensile strength values in the heat treated condition.

Analysing these results, it could be concluded that the compositions that preliminarily fulfil the criteria established for the preliminary selection of the alloys were the following: 5, 6, 15 and 16.

**WP2: Production of high temperature resistant alloys**

The objective of the second phase was therefore to focus the work and resources on the most promising alloys identified in the first phase, i.e. FC1 and FC2. The composition of these alloys was further detailed with inputs from the Topic Manager in terms of the need of providing corrosion resistance to the alloys and from Refial, the company in charge of producing the final batches that analysed the composition and established the compositional limits that could be reached by the industry keeping in mind the need of controlling the final cost of the alloys.

New batches were therefore produced in order to analyse other aspects that had not been approached during the first phase. Following the list of main aspects that were studied in the WP2 are listed:

• Formability. Creation of froming maps with the help of the Gleeble 3800 equipment and Garofalo´s equation.
• Thermal treatment optimization
• Castability tests.
• Study of recycling aspects

In the first task of WP2, around 25 kgs of each of the two alloys selected were prepared by Refial. These alloys were supplied as ingots of 10-12 kgs (Figure 10). From then the specimens needed for the conformability study of the materials were machined. It was also used to carry out the castability tests.


Figure 10. Aluminium ingots prepared by Refial

In order to produce the described alloys, REFIAL employed a pilot-scale gas-fired tilting rotary furnace, normally used to melt samples of secondary raw materials. The furnace metal capacity is around 25-40 kg, depending on the density of the raw materials to be melted and the amount of dross formed during the melting process. The following picture shows the furnace used to obtain the alloys.

Figure 11: Rotary furnace used to produce final batches of FC1 and FC2.

Once the raw materials were selected, the alloy composition is mathematically simulated using an Excel sheet taking into account the amount of each raw material to be added, the chemical characteristics of each raw material and the metallurgical yield obtainable in each case. As the metallurgical yield is influenced not only by the nature of each element but also by the melting equipment and process employed, it is usually specified based on empirical experience.

The next step of the process involves heating up the furnace and charging it with the calculated amounts of raw materials. The addition of the different raw materials follow a specific sequence depending on their melting point, their reactivity with furnace atmosphere and the time required to dissolve them in the aluminium matrix. As a first step, once the furnace is hot, the aluminium is charged in successive batches, as the furnace volume is not able to contain the total volume of the aluminium ingots. After heating up the furnace content and as the metal melts, further amount of aluminium may be added until a metal pool is obtained and the total amount of metal is fed. Over the metal pool dross is formed due to metal oxidation, which it is agitated manually helping the metal retained to reach liquid metal pool.

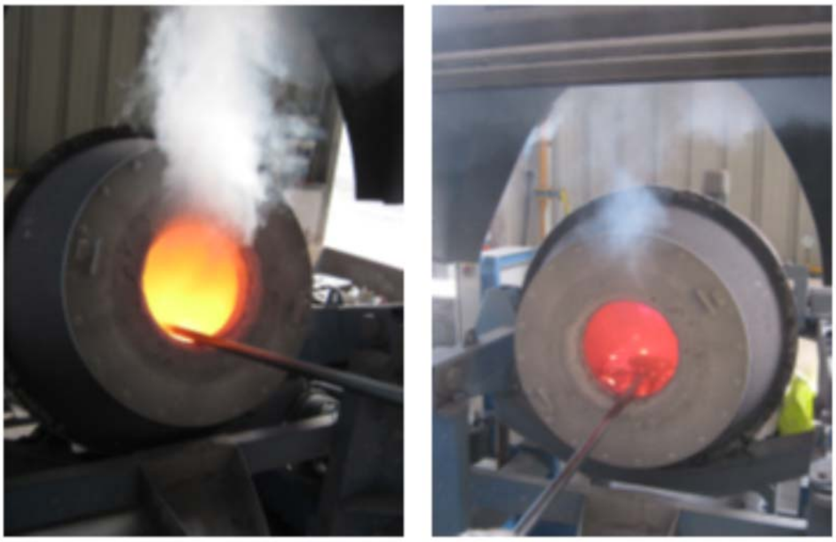

Figure 12: Production of the FC1 and FC2 aluminium batches

At this point, the slags are removed from the furnace and the less reactive, the higher melting point and the digestion resistant alloying elements (i.e., Cu, Ni, Fe, Mn, Si, Zn, Cr, Ti and Zr) are added over the metal pool, which is agitated manually to sink the alloying elements in the liquid aluminium pool. The furnace is then heated up during 30-45 minutes prior to take a sample for characterization.

As the metallic yield of the different alloying elements are interrelated in some cases, and are highly dependent on the process conditions, the metallic yield specified in the mathematical simulation is only a first approach to be corrected during the melting and alloying process taking and analyzing samples from the liquid metal pool. The sampling tool is immersed in the metal pool to get warmed up, and a first test sample is cast in the mould. This test sample is discarded (it is used to warm the mould) and a second sample is taken from the metal pool and cast in the mould. The test sample thus obtained is machined to get a dead-flat surface prior to its analysis in a spark optical spectrometer.

The spectrometer is verified and adjusted prior to the analysis of the test sample applying certified reference materials whose composition cover the alloying elements to be quantified in the sample. In this way, the accuracy of the results obtained are maximized.

The chemical composition of the test sample thus obtained and an estimation of the weight of the liquid metal pool is fed back in the mathematical simulation tool to estimate the amount of each alloying element to be added in order to get the desired alloy composition. In the case of the alloy number 2, FC2, used as example here, Ti, Zr, and Cr must be added in order to get into the tolerance range of each alloying element.

After the addition of alloying elements, a heating time (15-30 min) is applied in the furnace to ensure the solubilization of added elements. After that, a new test sample is taken and analyzed following the procedure mentioned above. In this way, the metal pool composition is adjusted in successive steps until each alloying element content falls inside the tolerance range.

Once the less reactive, higher melting point and digestion resistant elements are within tolerances, then the amount of remaining alloying elements (namely Mg and Sn) to be added are calculated and added to the furnace after retiring the slags of the liquid metal pool. These elements are burned easily in a gas fired furnace and therefore they must be added in the final stages to maximize their recovery and minimize the slag formation.

After heating up the furnace for 5 minutes, a new test sample is taken from the metal pool and chemically characterized. If the metal pool complies with the requirements, then it is cleaned manually with the proper tool and cast in preheated ingot moulds by tilting the furnace. Otherwise, further alloying steps must be applied until the alloy complies with specifications. It should be kept in mind that more time to get the alloy implies a lower overall yield as the metal tends to be oxidized in the gas fired tilting furnace.

In this way, it should be noted that Alloy FC1 is burned more easily in gas fired furnace than Alloy FC2, probably due to its lower copper content. Therefore, it is more difficult to obtain the former and lower quantities of metal alloy results. Several batches of Alloy number 1 have been done in order to get the expertise necessary to fulfill the required alloy quantity in one single batch.

**Forming maps. Introduction**

A common method for controlling metal forming processes is by means of Forming Maps. Forming Map DMLE (mechanical stability) is a two-dimensional representation of Strain rate vs Temperature in which a stability criterion to formation of deformation bands that may give rise to the appearance of cracks is applied. Therefore a parameter related to the probability of appearance of cracks is calculated in the map obtaining different stability bands.

To construct the maps, it is necessary to start from a constitutive equation (Garofalo equation) that relates the experimental variables of the material: stress, temperature and strain rate. Garofalo equation can be calculated from the results obtained in the physical simulation of thermomecanical conditions of the material (analysis of the deformation behaviour of the studied material).

Particularly in this study the physical simulation of the studied materials has been performed by means of hot axisymmetric compression tests, performed in GLEEBLE® 3800 System of TECNALIA. Stress-strain curves of the studied aluminium alloys are obtained at different strain rates and temperatures.

Compression test specimens were lengthwise machined from the delivered rods: Cylindrical samples, of 10 mm diameter and 15 mm in length.


Figure 13. Compression test specimens machined from delivered aluminums

Forming Map DMLE (mechanical stability) is a two-dimensional representation of Strain rate vs Temperature in which a stability criterion to formation of deformation bands that may give rise to the appearance of cracks is applied. Therefore a parameter related to the probability of appearance of cracks is calculated in the map obtaining different stability bands.

To construct the maps, it is necessary to start from a constitutive equation (Garofalo equation) that relates the experimental variables of the material: stress, temperature and strain rate. Garofalo equation is calculated from the results obtained in the physical simulation of thermomecanical conditions of the material by means of hot axisymmetric compression tests, performed in GLEEBLE ® 3800 System of TECNALIA. Stress-strain curves of the studied aluminium alloys are obtained at different strain rates and temperatures.

Applying the2nd Liapunov criterion (stability criterion to formation of deformation bands that may give rise to the appearance of cracks) as a function of strain rate and temperature the Forming Map DMLE is obtained for the aluminium.

Temperature and strain rate windows

The process windows (Temperature and strain rate windows) are defined from previous calculation from extrusion process data in order to construct the maps. Temperature data are directly available from the extrusion process data. Strain and strain rate have to be calculated from the extrusion process data.

The Liapunov function [$\delta m$ / $\delta \ln\varepsilon'$] is calculated from $\ln\varepsilon'$ = -0,7 ($\varepsilon'$= 0,50 s-1) to $\ln\varepsilon'$ = 0,3 ($\varepsilon'$= 1,35 s-1) and from T = 420ºC to T = 475ºC. The Forming Map DMLE obtained for the FC1 alloy is shown in next Figure.

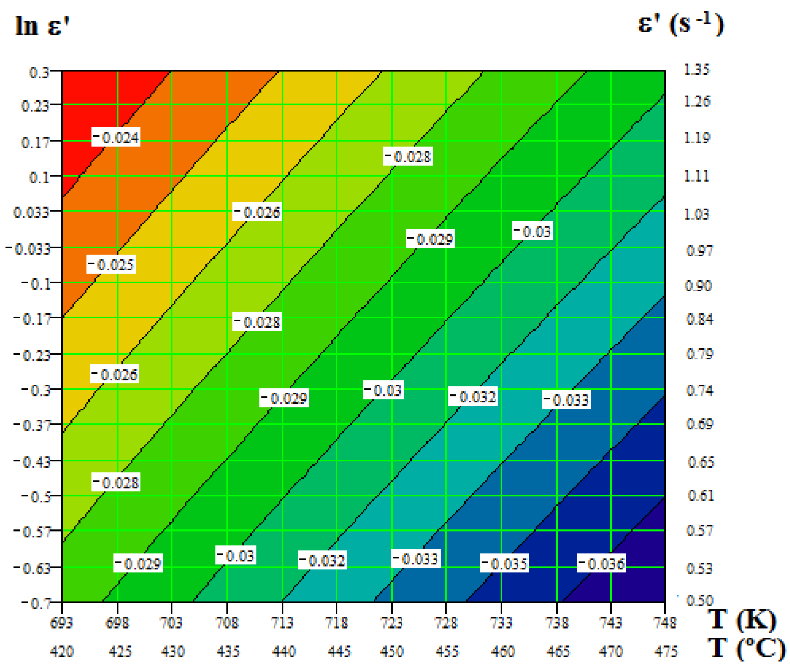


Figure 14. Forming Map DMLE – FC1 alloy

In the case of the alloy FC2 The Liapunov function [δm / δlnε'] is calculated from lnε' = -0.7 (ε'= 0,5 s-1) to lnε' = 0,3 (ε'= 1,35 s-1) and from T = 420ºC to T = 475ºC. The Forming Map DMLE obtained is shown in next Figure.

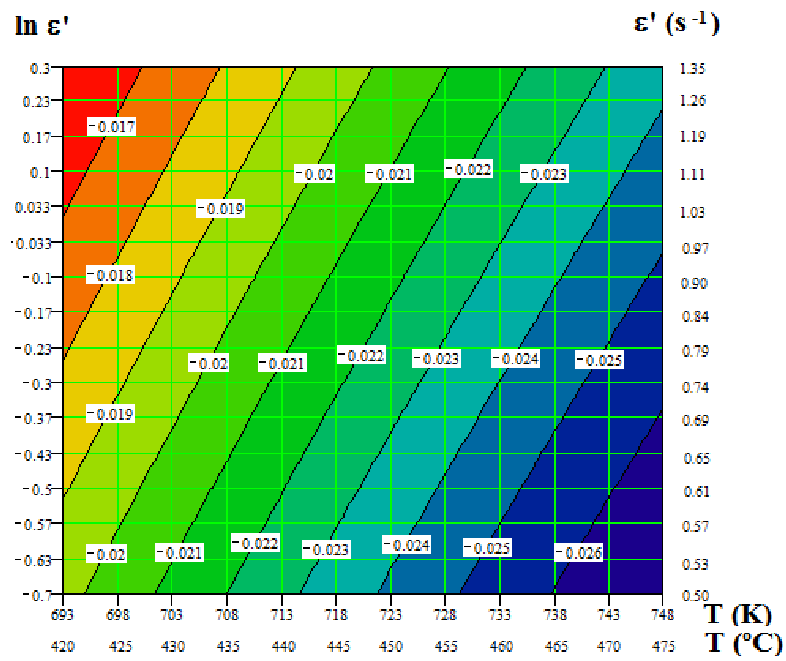


Figure 15. Forming Map DMLE – FC2

## Study of recycling aspects

Both alloys, but especially the FC2 alloy, are highly alloyed aluminium alloys, being Mg and Ni the common alloying elements and differing in Cu, Fe and Mn content. In both cases, they include Zr and Cr as grain refiners. Ti is also added to the second alloy.

These alloys continue under development and they have not been classified between wrought or cast alloys yet. During the project they have been extruded but castability tests are being performed as well.

In order to evaluate the reciclability of these alloys, the aluminium recyclability indexes approach is considered. The values of calculated ARI indexes and qualified RPI indexes for the mentioned alloys are shown in the following table:

| Alloy | ARI | RPI |
|---|---|---|
| A1 | 91,05 | L |
| A2 | 86,60 | U |

Table 4. ARI and RPI values for the developed alloys.

According to the ARI values both alloys exhibit a low recyclability. This is especially true for alloy FC2, due to its very high alloying content, related mainly to Cu.

The RPI qualification is not favourable for recycling since the amplitude of the tolerance ranges for different alloying elements are relatively narrow. The alloy number 1 is penalized by its low copper content, which will be higher in those alloys that may be mixed with (e.g., 2024 or similar). Alloy 2 shows relative low tolerance to Fe. In both cases, the presence of tin is harmful to most wrought and cast alloys obtainable.

It should be emphasized that the main alloying element for high temperature application, i.e., nickel, is not used in common aircraft or other commercial alloys, as should be noted from the composition presented in previous section of this work. Those recycling schemes that lead to obtain alloy mixtures will be jeopardized by the presence of this “exotic” element, which may be considered a contaminant. In the case of alloy number 1, the high Fe and Mn contents may cause contamination of the whole mixture as these elements cannot be removed during refining and usually reduce ductility of the alloys.

Selective dismantling of the components formed with this alloys will enable the effective recycling of both alloys. In this case, however, it should be kept in mind that successive remelting steps will have a deleterious effect on fatigue resistance and, therefore, the recycled alloy may be applied in non-critical applications.

## Thermal treatment optimization

The T6 thermal treatment consists of three phases:

- Solution heat treating. The alloy is heated to a temperature that is high enough to put the soluble alloying elements in solution.
- Rapidly quenching to a lower temperature. The alloy is quenched to a lower temperature to keep the alloying elements trapped in solution.

- Artificial ageing. The alloy is heated to an intermediate temperature so that the alloying elements trapped in solution can precipitate to form a uniform distribution of very fine particles that harden the aluminium matrix.

As it would be a very large process to study all the parameters of this kind of thermal treatment, it was decided to fix the parameters of the solution n heat treating and quenching steps and study only the parameters of the artificial ageing. So, it was decided to solution heat treat all the samples at 530ºC during five hours and quench them in water to room temperature.

In order to define the ageing process optimization scheme, a bibliographical study was first carried out. As a general conclusion, it was seen that usually a peak hardness value is obtained for each ageing temperature, being this peak hardness higher when the temperature is reduced. However, at lower temperatures the peak hardness is reached at longer times, thus increasing the ageing process duration.

Searching for information about thermal treatment of Al-Cu alloys with similar compositions to the ones to be studied, the ageing process optimization scheme was defined, for both the low-copper alloy (Table 5) and high-copper alloy (Table 6).

For each alloy four different ageing temperatures were selected. For FC1, temperatures from 175ºC to 250ºC were selected, while for FC2, lower temperatures ranging from 150ºC to 225ºC were selected, as the increase in the copper content reduce the temperature needed to obtain high hardness values.

| FC1 | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Temperature | Time (in hours) | | | | | | | | | | | | | |
| | 0,1 | 0,5 | 1 | 2 | 4 | 8 | 12 | 16 | 20 | 25 | 30 | 36 | 48 | 72 |
| 150 | | | | | | | | | | | | | | |
| 175 | | | | | | | x | x | x | x | x | x | x | x |
| 200 | | | | | | x | x | x | x | x | x | x | x | |
| 225 | | | | x | x | x | x | x | x | x | x | | | |
| 250 | | x | x | x | x | x | x | x | x | | | | | |

Table 5. Ageing process optimization scheme for FC1.

| FC2 | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Temperature | Time (in hours) | | | | | | | | | | | | | |
| | 0,1 | 0,5 | 1 | 2 | 4 | 8 | 12 | 16 | 20 | 25 | 30 | 36 | 48 | 72 |
| 150 | | | | | | x | x | x | x | x | x | x | x | |
| 175 | | | | | x | x | x | x | x | x | x | x | | |
| 200 | | | x | x | x | x | x | x | x | x | | | | |
| 225 | x | x | x | x | x | x | x | x | | | | | | |
| 250 | | | | | | | | | | | | | | |

Table 6. Ageing process optimization scheme for FC2.

The selection of the most suitable heat treatment parameters was based on the microhardness results. The ageing temperature and time needed to reach the maximum hardness were selected as the optimum parameters for the heat treatment to apply to the samples to be mechanically characterized in WP3 of the project.

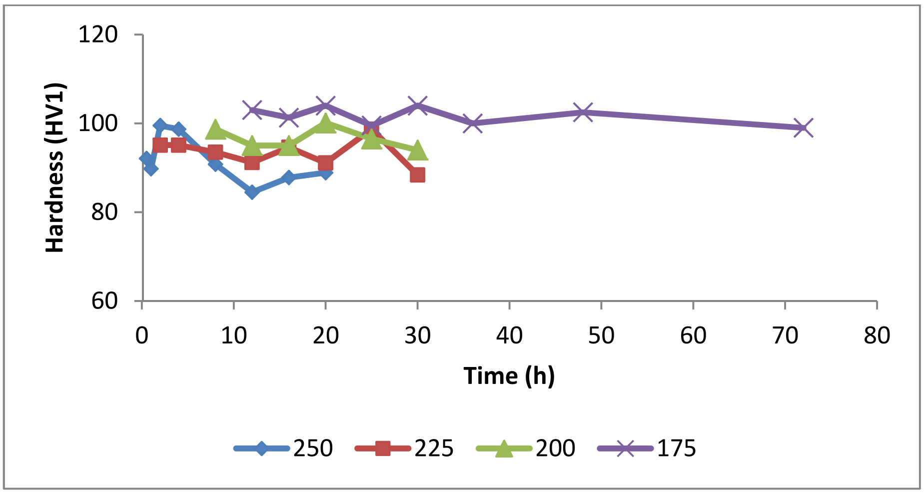


Figure 16. Ageing curves at different temperatures for FC1.

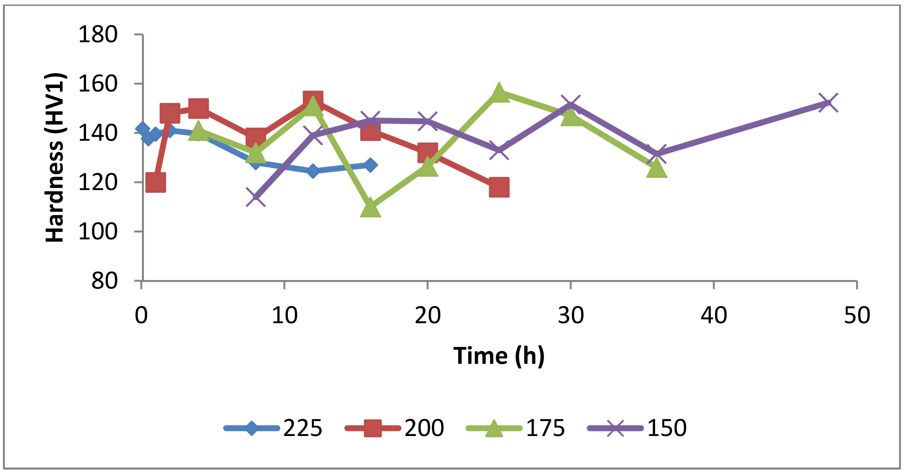


Figure 17. Ageing curves at different temperatures for FC2.

The analysis of the results obtained in the optimization of the ageing process lead to the conclusion that the highest hardness is obtained at a temperature of 175ºC for both alloys. The ageing time to reach the higher hardness value is different depending on the alloys: the high copper alloy reach this peak hardness at 175ºC at a shorter time (25 hours) than the low copper content alloy (30 hours).

## WP3: Characterization

After the optimization of the thermal treatment the last batches of the two alloys were prepared, extruded and thermally treated in order to carry out the analysis of the microstructure, mechanical properties and the study of the performance of the alloys at high temperatures. Following the main results and conclusions obtained are detailed.

### Mechanical and metallurgical characterization

The main two objectives of the work carried out that are explained in this deliverable are the following:

- To characterize mechanically the two alloys selected at the end of WP1 in the as-extruded + heat treated condition. The mechanical characterization has included tensile testing and creep rupture testing.
- To characterize the microstructure of the two alloys selected at the end of WP1 in the as-extruded and extruded + heat treated conditions. This characterization has included optical microscopy, scanning electron microscopy (SEM), electron dispersive spectroscopy (EDS) and X-ray diffraction (XRD).

Table 7 shows a summary of the mechanical testing that was agreed to be performed between the Topic Manager and the partners of the project.

•

| Test | Temperature (ºC) | Condition of samples | Responsible | Tested specimens |
|---|---|---|---|---|
| Tensile | 20 | Extruded + HT | Tecnalia | 5 |
| | 250 | Extruded + HT | Tecnalia | 5 |
| | 250 | Extruded + HT + Aged | Tecnalia | 5 |
| Creep | 250 | Extruded + HT | Tecnalia | 6 |

Table 7. Mechanical testing performed to each alloy

Ten specimens **¡Error! No se encuentra el origen de la referencia.**from each composition were machined for tensile testing. Five of these samples were tested at room temperature and the other five at 250ºC in order to compare the obtained data with the results from the as-extruded and extruded + heat treated samples before the optimization of the heat treatment. These results were obtained during the characterization carried out in WP1.

Six specimens from each composition were machined for creep testing. For this testing, five different stress levels were defined for each composition.

Table8 shows a summary of the metallurgical characterization performed to the extruded samples before and after the heat treatment.

| Technique | Samples | Analysis |
|---|---|---|
| Optical Microscopy | As-extruded and extruded + HT | 2 micrographs at x50 and x400 magnifications |
| SEM | As-extruded and extruded + HT | 2 micrographs at x500 and x2000 magnifications |
| XRD | As-extruded and extruded + HT | Analysis of the phases |
| EDS | As-extruded and extruded + HT | Composition of the different phases |

Table 8. Summary of metallurgical characterization

A section of 17x3mm was cut from the head of the tensile specimen and it was polished and etched in order to see its microstructure in the OM and the SEM. Two micrographs were obtained from the optical microscope at different magnifications (x50 and x400). In the SEM, the different phases were identified and their composition was analyzed by EDS. Two micrographs were obtained at x500 and x2000 magnifications. Finally, an XRD was performed to the samples to complete the information about the phases that appear in each of the samples.

In the following tables the tensile results of all the specimens tested are shown.

| Ref | FC1-1 | FC1-2 | FC1-3 | FC1-4 | FC1-5 | Av. | SD |
|---|---|---|---|---|---|---|---|
| Ys (MPa) | 276 | 234 | 268 | 241 | 250 | 253.80 | 17.78 |
| UTS (MPa) | 309 | 303 | 330 | 306 | 339 | 317.40 | 16.07 |
| ε (%) | 3.6 | 6.6 | 5.2 | 5.8 | 11.3 | 6.50 | 2.90 |

Table 9. Tensile testing results for FC1 at room temperature.

| Ref | FC1-6 | FC1-7 | FC1-8 | FC1-9 | FC1-10 | Av. | SD |
|---|---|---|---|---|---|---|---|
| Ys (MPa) | 177 | 186 | 201 | 196 | 201 | 192.2 | 10.47 |
| UTS (MPa) | 222 | 222 | 234 | 231 | 228 | 227.40 | 5.37 |
| ε (%) | 15.6 | 14.7 | 11.4 | 16.2 | 16.9 | 14.96 | 2.15 |

Table 10. Tensile testing results for FC1 at 250ºC

| Ref | FC2-1 | FC2-2 | FC2-3 | FC2-4 | FC2-5 | Av. | SD |
|---|---|---|---|---|---|---|---|
| Ys (MPa) | 336 | 329 | 331 | 334 | 353 | 336.60 | 9.56 |
| UTS (MPa) | 434 | 431 | 430 | 433 | 439 | 433.40 | 3.51 |
| ε (%) | 9.7 | 9.6 | 10.5 | 9.0 | 8.0 | 9.36 | 0.93 |

Table 11. Tensile testing results for FC2 at room temperature.

| Ref | FC2-6 | FC2-7 | FC2-8 | FC2-9 | FC2-10 | Av. | SD |
|---|---|---|---|---|---|---|---|
| Ys (MPa) | 234 | 213 | 204 | 241 | 249 | 228.20 | 19.02 |
| UTS (MPa) | 260 | 264 | 268 | 268 | 283 | 268.60 | 8.71 |
| ε (%) | 13.9 | 5.3 | 7.4 | 5.8 | 9.6 | 8.40 | 3.50 |

Table 12. Tensile testing results for FC2 at 250ºC.

Table 13 shows the results of the creep rupture testing performed to samples of the FC1 alloy and Table 13. Creep rupture testing results for FC1 at 250ºC.

14 shows the results of the testing of FC2 alloy.

| Ref. | Stress (MPa) | Time to rupture (h) |
|---|---|---|
| FC1-1 | 150 | 0.1 |
| FC1-2 | 150 | 0.1 |
| FC1-3 | 120 | 5.9 |
| FC1-4 | 105 | 9.3 |
| FC1-5 | 90 | 38.1 |
| FC1-6 | 135 | 1.0 |

Table 13. Creep rupture testing results for FC1 at 250ºC.

| Ref. | Stress (MPa) | Time to rupture (h) |
|---|---|---|
| FC2-1 | 180 | 0.3 |
| FC2-2 | 180 | 0.2 |
| FC2-3 | 140 | 4.9 |
| FC2-4 | 120 | 17.2 |
| FC2-5 | 100 | 93.1 |
| FC2-6 | 160 | 1.7 |

Table 14. Creep rupture testing results for FC2 at 250ºC

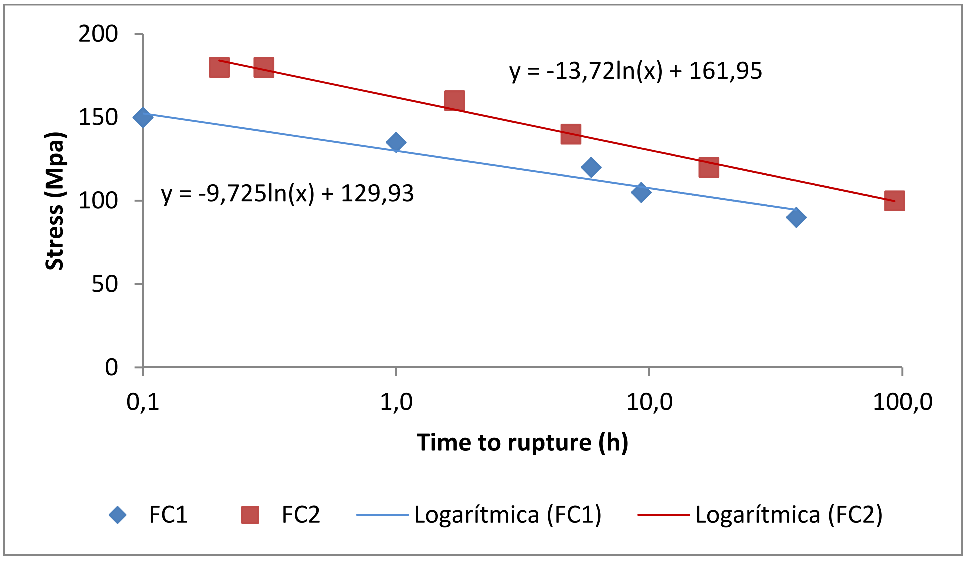


Figure 18. Creep to rupture for FC1 and FC2 at 250ºC.

In general, mechanical properties (tensile and creep properties) of FC2 alloy are better than those for FC1 alloy in all the studied conditions. For the low-copper content alloy, the strength at 250ºC that could not be improved when the conventional heat treatment was applied in the first phase of the project, was increased around 50% after the optimization of the heat treatment. The reason of this improvement could be found in the precipitation of different particulates, not only $Al_2Cu$ particulates but also $Mg_2Si$ particulates. A yield strength of 192MPa and a tensile strength of 227MPa are reached in the FC1 alloy when it is subjected to the heat treatment with the parameters selected after the work performed in WP2.

For the high copper content alloy, the mechanical resistance at elevated temperature was improved when the conventional heat treatment was applied, but after applying the optimized heat treatment the resistance is further improved and comparing yield strength and tensile strength values with those for the as-extruded condition, they are more than doubled reaching values of 228MPa in the yield strength and 268MPa in the tensile strength. The main reason of this increase is the precipitation of very fine Al2CuMg particulates that should be distributed uniformly along the matrix, although they couldn't be identified in the SEM due probable to their small size.

**Prediction of the ageing behavior**

The main objective of the task related to the prediction of the behaviour of the alloys at high temperatures was to analyse the effect that holding the alloys during 1000 hours at a temperature of 250ºC may have on the microstructure and the tensile properties.

So, one small cylinder of around 100mm height of each alloy was introduced in an oven and the temperature was set in 250ºC. After reaching this temperature, the alloys were maintained at that temperature during nearly 42 days (1000 hours).

Five tensile specimens were machined from each cylinder and they were tested at 250ºC in order to compare the results with the non-aged samples tested previously.

In the following tables the tensile results of all the specimens tested are shown.

| Ref | FC1-11 | FC1-12 | FC1-13 | FC1-14 | FC1-15 | Av. | SD |
|---|---|---|---|---|---|---|---|
| Ys (MPa) | 109 | 108 | 102 | 115 | 112 | 109.2 | 10.47 |
| UTS (MPa) | 128 | 128 | 126 | 133 | 129 | 128.8 | 2.59 |
| ε (%) | 28.5 | 41.0 | 39.5 | 29.0 | 28.5 | 33.3 | 6.37 |

Table 15. Tensile testing results for aged FC1 at 250ºC

| Ref | FC2-11 | FC2-12 | FC2-13 | FC2-14 | FC2-15 | Av. | SD |
|---|---|---|---|---|---|---|---|
| Ys (MPa) | 116 | 105 | 100 | 96 | 108 | 105.0 | 7.68 |
| UTS (MPa) | 140 | 132 | 128 | 123 | 135 | 131.6 | 6.5 |
| ε (%) | 25.0 | 28.0 | 21.0 | 24.5 | 22.0 | 24.1 | 2.75 |

Table 16. Tensile testing results for aged FC2 at 250ºC.

After studying the microstructure by means of optical microscopy and SEM, it could be concluded that holding of the developed alloys at a temperature of 250ºC during 1000 hours produces a modification of the microstructure. In both alloys it can be appreciated that the $Al_2CuMg$ phase is located in the grain boundaries forming a network. This phase, that couldn't be appreciated in the samples before the ageing process seems to have been formed due to the joining and coarsening process of the precipitated particulates that were previously dispersed uniformly along the matrix. Due to the higher amount of copper of the high copper content alloy, this phase is present in a higher amount in this alloy than in the low copper content one.

The modification of the microstructure leads to traduced in a loss of mechanical properties. For FC1, both yield and tensile strength are reduced around a 45% after the ageing process, while for FC2 the reduction is even higher, around a 55%.

**WP4: Technical, economical and environmental analysis**

The main result of the ALT project is that two aluminium alloys for high temperature applications have been developed that present a good balance of properties and seem to reach the established level of properties. Even so it is deemed that further tests would be required in order to fully develop the alloy. The analysis of the microstructure and the influence of thermal treatment and holding the alloys at high temperatures for long periods of time should be further understood in order to optimise the composition of the alloy and make sure of its long term performance under high temperature service conditions.

The main conclusions that may be drawn from the analysis of the measurement of properties of developed alloys are that in general, mechanical properties (tensile and creep properties) of FC2 alloy are better than those for FC1 alloy in all the studied conditions. For the low-copper content alloy (FC1), the strength at 250ºC was increased around 50% after the optimization of the heat treatment. The reason of this improvement could be found in the precipitation of different particulates, not only Al2Cu particulates but also Mg2Si particulates. A yield strength of 192MPa and a tensile strength of 227MPa are reached in the FC1 alloy when it is subjected to the heat treatment with the parameters selected after the work performed in WP2.

For the high copper content alloy (FC2), the mechanical resistance at elevated temperature was improved when the conventional heat treatment was applied, but after applying the optimized heat treatment the resistance is further improved and comparing yield strength and tensile strength values with those for the as-extruded condition, they are more than doubled reaching values of 228MPa in the yield strength and 268MPa in the tensile strength. The main reason of this increase is the precipitation of very fine Al2CuMg particulates that should be distributed uniformly along the matrix, although they couldn't be identified in the SEM due probable to their small size.

Both alloys seem to present a good balance of properties and it is deemed that further studies should be made to complete the characterization of the alloys and to complete some tasks that have not been made clear completely in this project, e.g. Is the composition of the alloys optimized, may it be further adjusted to improve properties at high temperatures?. Which is the exact correlation between process parameters/microstructure and properties for these alloys? Is the thermal treatment fully optimized?...

***Regarding economical aspects*** one of the main features of the project is that the use of conventional and therefore not high cost alloying elements and processes were sought for the development and production of wrought aluminium alloys for high temperature applications.

The economic evaluation is referred to a melting and alloying process in two steps. In the first step, the aluminium charge is melted using a rotary furnace and in the second step the molten metal is alloyed in a reberveratory furnace. This approach is employed extensively when obtaining aluminium alloys from secondary raw materials because it combines the versatility and efficiency of the rotary furnace to manage complex raw materials with the stability and homogenising action (when properly stirred) of the reverberatory furnaces to obtain top quality allows.

Figure 19. Rotary and reverbatory furnaces used for the production of the final batches of the developed alloys.

In the case of obtaining alloys from primary aluminum ingots, the melting and alloying steps may be performed in a single furnace, usually a reverberatory furnace. However, the thermal efficiency during melting of these kind of furnaces is worse, although this fact may no be as relevant when the cost of the raw materials to produce special alloys is high enough relative to the energy costs.

Also, it should be noted that the type of furnace heating element may be dependent on the amount of the alloys to be produced. For low quantities, i.e., below 2-5 t per batch, the electric heated crucible furnaces may be a good approach, whereas for bigger production capacities (i.e. 20-25 t/batch) gas-fired furnaces are usually the best choice.

As mentioned above, the cost analysis performed is based on two step gas fired furnaces, considering as raw materials primary metals or specific mixtures for alloying. No further refining (degassing, filtering) and/or processing steps have been considered in the analysis cost, although they should be considered if the alloy should be casted in the form of bars or slabs to be processed by forging or extrusion. Thus the discussion about the cost of the alloys under development in constrained to the production of ingots for melting.

The analysis includes all the costs involved during normal production of aluminium alloys: personnel costs, consumables (fluxes), maintenance costs, energy costs, equipment amortization…, including those costs related to the wastes generated during the production (slags, filter dust).

It must be kept in mind that the price estimation for each alloy is highly dependent on the international demand/supply balance of some of the raw materials employed in its production. Thus, the prime of each alloy must be considered an approximate value estimated with the conditions of the market at the moment of writing this report. The following table show the marketable price of each alloy in a first approach, which should be corrected when considering the industrial production under demand.

| Alloy | Price €/t |
|---|---|
| Alloy 1 | 3.650 |
| Alloy 2 | 3.950 |

Table 17. Estimated cost of the developed alloys.

From the cost analysis, it is derived that the main cost is, by far, the raw materials costs, accounting more than 90 % of the totals cost. Among the raw materials, primary aluminium accounts approximately half of the total material costs for both alloys and, therefore, its international pricing will have a big influence in the final price of the considered alloy selling price.

In both cases, nickel and zirconium pricing may have a significant contribution to the final selling price. In the case of nickel, primary metal has been considered for alloying, being its price fixed through international trade. In the case of zirconium, its addition is considered through the use of alloying tablets, where each tablet contains a precise weight of high purity alloying element, the balance being aluminium or a mixture of aluminium plus selected Na free, non-hydroscopic fluxes to accelerate dissolution and recovery. As a manufactured product, its final price is dependent on the formulation elements international pricing but also on the economies of scale.

In the case of alloy FC2, copper related cost should be mentioned because the relative amount of this alloying element in quite significant and the international pricing is relevant.

The mentioned elements accounts around the 90-95 % of the raw materials cost of the developed alloys. Energy, personnel, maintenance and other costs have little impact on the final price of the alloys in the analysis performed, but their relative importance may be increased significantly if the amounts of the alloys to be produced are under the threshold of 25 ton per batch. Also, further processing steps required depending on final application (i.e. refining, degassing, filtering…) are not included in the alloys pricing.

***Regarding environmental aspects*** In order to evaluate the reciclability of these alloys, the aluminium recyclability indexes approach is considered. The values of calculated ARI indexes and qualified RPI indexes for the mentioned alloys are shown in the following table:

| Alloy | ARI | RPI |
|---|---|---|
| A1 | 91,05 | L |
| A2 | 86,60 | U |

Table 18: ARI and RPI data of the developed alloys

According to the ARI values both alloys exhibit a low reciclability. This is especially true for alloy FC2, due to its very high alloying content, related mainly to Cu.

The RPI qualification is not favourable for recycling since the amplitude of the tolerance ranges for different alloying elements are relatively narrow. The alloy number 1 is penalized by its low copper content, which will be higher in those alloys that may be mixed with (e.g., 2024 or similar). Alloy FC2 shows relative low tolerance to Fe. In both cases, the presence of tin is harmful to most wrought and cast alloys obtainable.

It should be emphasized that the main alloying element for high temperature application, i.e., nickel, is not used in common aircraft or other commercial alloys, as should be noted from the composition presented in previous section of this work. Those recycling schemes that lead to obtain alloy mixtures will be jeopardized by the presence of this “exotic” element, which may be considered a contaminant. In the case of alloy number 1, the high Fe and Mn contents may cause contamination of the whole mixture as these elements cannot be removed during refining and usually reduce ductility of the alloys.

Selective dismantling of the components formed with this alloys will enable the effective recycling of both alloys. In this case, however, it should be kept in mind that successive remelting will have a deleterious effect on fatigue resistance and, therefore, the recycled alloy may be applied in non-critical applications.

In summary the main conclusions drawn for the analysis of the obtained results are as follows:

- Two alloys with different compositions have been developed in the project with success: FC1 or low copper and FC2 or high copper content alloys.

- The alloys belong to the Al-Cu series, present low and high copper contents and the presence of Nickel and magnesium as the main alloying elements.

- Technical properties of the alloys seem to comply with the established validation criteria for the envisaged application. This should be confirmed with longer ageing treatments, corrosion tests and fatigue tests though.

- It is considered that there is still some margin to improve the performance of the alloys. Small changes in the composition, ageing mechanisms and adjusting the magnesium content or cold drawing are possibilities for further improvement.

- The alloys present rather good castability, comparable to other Al-Cu alloys and it could be worth analysing their potential as casting alloys

- Formability tests with the Gleeble test equipment present good results

- The T6 thermal treatment has been optimized. Mechanical properties get improved by more than 40%. The possibility of applying cold drawing and further optimizing the thermal treatment should be considered in future developments.

- The cost analysis provides an estimation of the cost of both alloys (FC1 and FC2) as 3.65 €/Kgs. and 3.95 €/Kgs. respectively.

- The recyclability and environmental aspects of the alloys do not differ much from other conventional Al-Cu alloys

## Potential impact

### Introduction

The project "Formulation and Characterization of New Aluminium Alloys Produced by Ingot Metallurgy for High Temperature Applications (250ºC)" has been devoted to the research and development of wrought aluminium alloys that may be used in future air conditioning systems of commercial airplanes that have to work at temperatures of up to 250ºC. The project has been successful from the technical point of view and two different alloys have been developed that comply with the established requirements. Nevertheless the cost analysis results in cost values higher than those of the alloys currently used in the foreseen application and therefore new applications or new designs requiring better performance at high temperatures should be identified to fully exploit the obtained results.

Following the main conclusions of the project are listed:

- Copper and magnesium improve the strength at all the different conditions studied and at both room temperature and high temperature. Nickel increases the strength in non-heat treated conditions, while after the heat treatment it seems that the Ni presence is deleterious for the mechanical properties of the alloys. Manganese seems to have a positive effect only on the elevated temperature strength and not on the room temperature strength. Titanium has also a positive effect on the elevated temperature strength. And silicon is the most deleterious element according to the results obtained in this project, because its presence produces a reduction in the mechanical properties in nearly all the conditions.
- In general, mechanical properties (tensile and creep properties) of FC2 alloy are better than those for FC1 alloy in all the studied conditions.
- For the high copper content alloy, the mechanical resistance at elevated temperature got more than doubled reaching values of 228MPa in the yield strength and 268MPa in the tensile strength after the application of the optimized heat treatment. The main reason of this increase is the precipitation of very fine $Al_2CuMg$ particulates that are distributed uniformly along the matrix.
- Holding the developed alloys at a temperature of 250ºC during 1000 hours produces a modification of the microstructure. In both alloys it can be appreciated that the $Al_2CuMg$ phase is located in the grain boundaries forming a network. This phase, that could not be seen in the samples before the ageing process seems to have been formed due to the joining and coarsening process of the precipitated particulates that were previously dispersed uniformly along the matrix. This phase is present in a higher amount in this alloy than in the low copper content one.
- The modification of the microstructure is traduced in a loss of mechanical properties. For FC1, both yield and tensile strength are reduced around a 45% after the ageing process, while for FC2 the reduction is even higher, around a 55%.

- Tensile properties at high temperature of both alloys are very similar after the ageing process and slightly higher than most of the commercial alloys.
- Creep properties are better for high copper content alloy and this alloy has a creep behaviour very similar to that of the AA2618.

The importance of the heat treatment to increase the mechanical properties at high temperatures has been confirmed, so further work to do in the future could be based on studying more in depth the heat treatment parameters, not only the ageing T and times but also the solutionizing parameters, or even trying different treatments such as some cold drawing that is known to be successful for improving the properties in some alloys.

**Socio-economic impact**

The Clean Sky Joint Technology Initiative contributes to the research challenges that are necessary to reach the environmental goals set by the ACARE (Advisory Council for Aeronautics Research en Europe) to be reached in 2020. These are:

1. 50% reduction of $CO_2$ emissions through drastic reduction of fuel consumption.
2. 80% reduction of $NO_x$ (nitrogen oxide) emissions.
3. 50% reduction of external noise.
4. A green product life cycle: design, manufacturing, maintenance and disposal/recycling.

The ALT project has been devoted to the development of light wrought alloys that could be used in the aeronautical market and that would approach all hose aspects listed above. Furthermore the developed alloys have preliminarily shown that they might even used for casting applications. The castability tests performed on themhas shown that they present better properties than other high temperature casting alloys such as 2618.

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The development of wrought aluminium alloys with the capability of withstanding higher temperatures than the current alloys (the goal is to reach up to 200-250ºC) may allow the development of many different components that are presently produced with heavier alloys and even with ferrous materials. The economical impact of this development is huge and difficult to measure. The developed alloys could find applications in many different industrial sectors in which light materials that may withstand 250ºC are sought. This range of temperatures is out of the scope of technical polymers with affordable costs and both the aeronautical and automotive sectors are investing huge amounts of resources in this kind of developments. The automotive sector needs light materials for under the hood applications such as pistons, camrods, cylinderheads for diesel engines as well as break discs and drums for future new designs that will have to be created to increase the efficiency of the motor and drivetrain components. The development of the electrical vehicle is also a huge potential market for these alloys.

The aeronautical and space sector is also another large potential market for high temperature aluminium alloys in components as all the forecasts available confirm the growth expectations of the sector and there are many applications for the aluminium alloys to be developed in the project.

The degree of development of the two alloys on which the work of the project has been focused is high but further works would be required to identiy the final applications for them , mainly so in the case of casting components as the project was basically focused on the development of wrought alloys.Following the main aspects to be studied are detailed:

Cost. A preliminary cost analysis has been made that shows that the developed alloys are 40-50% more expensive than other alloys currently used in applications of up to 250ºC. This is mainly due to the large amount of nickel in the alloys. This content could be adjusted and decreased in further developments.

Performance. More additional data of the performance of the alloys would be required before the alloys may substitute currently used materials in existing applciations. The behaviour in casting processes, optimisation of thermal treatment to further increase the obtained properties, adjustment of the composition, corrosion behaviour and fatigue properties are aspects that should be further analysed.

The successful development of the project would have an indirect impact in societal and environmental issues, The impact of the introduction of advanced light alloys in transport applications in Europe has been deeply analysed and discussed in the last 20 years and this project directly approaches the conclusions of such approaches related to improve sustainability of the European industry, protect the environment and comply with CO2 emissions limits and protect the European industrial employment rate.

**Dissemination activities and exploitation of results**

Regarding dissemination activities so far the main action has been related to the presentation of the work in the 71st World Foundry Cogress hld in Bilbao in May 2014.

In the first period of the project two abstracts were prepared and submitted to two different events following the previously described procedure. The first one was submitted to the Greener Aviation conference organised in Brussels to show the results of Cleansky projects but it was not accepted. The second abstract was sent to the organizing committee of the World Foundry Congress to be held in May 2014 in Bilbao. This abstract was accepted and the subsequent article was also accepted and has been included in to the official proceedings book of the conference. Furthermore the work was presented with an oral presentation.

During the final technical meeting the consortium agreed on the following two dissemination actions:

Presentation of a poster providing a general overview of the work and main results in the 3rd Industrial Workshop of the KMM_VIN virtual institute of the knowledge based multifunctional materials to be held in Dresden in 3-4 November 2014. This poster has already been accepted.

Preparation of two articles to be submitted to ISI scientific journals. These works will be prepared ion the second half of 2014 and published in 2015. Some of the aspects that could be included in this work are following listed:

● Design of experiments approach and preliminary results obtained with the 24 alloys.

● Thermal treatment optimisation approach and results. Comparison of high Copper content and low copper content alloys performance with thermal treatments.

● Extrusion. Influence of extrusion on the mechanical properties. Comparison of as cast and as extruded results.

● Properties of the alloys at room temperature.

● Microstructural aspects. Identification of phases and their role in the performance of the alloys.

● Influence of the 1000 hours treatment at 250ºC on the alloy properties and microstructure.

● Formability aspects of the developed alloys.

Regarding exploitation activities the main partner interested in exdploitation issues is Refial S.A., as producer and supplier of secondary aluminium alloys. This company had not any previous experience in the development of wrought alloys and the project has make it possible to them to access to this market.

The main results of the project are presented in table 19

| Type of Exploitable Foreground | Description of exploitable results | Exploitable products or measure | Patents or other IPR exploitation (licenses) | Owner & other Beneficiary involved |
|---|---|---|---|---|
| Ownership | Formulation for wrought Al alloys for high T applications | Formulation | Patent | TECNALIA, INATEC & REFIAL |
| Ownership | Formulation for casting Al alloys for high T applications | Formulation | Patent | TECNALIA, INATEC & REFIAL |

Table 19: Summary of the main exploitable results

It is deemed that the actual exploitability of these alloys will depend on the economic aspects. The cost analysis carried out signalles that the raw material, i.e. the master alloys used to produce the alloys represent up to 90% of the total cost of the alloy.

The analysis includes all the costs involved during normal production of aluminium alloys: personnel costs, consumables (fluxes), maintenance costs, energy costs, equipment amortization…, including those costs related to the wastes generated during the production (slags, filter dust).

It must be kept in mind that the price estimation for each alloy is highly dependent on the international demand/supply balance of some of the raw materials employed in its production. Thus, the prime of each alloy must be considered an approximate value estimated with the conditions of the market at the moment of writing this report. The marketable price of each alloy in a first approach which should be corrected when considering the industrial production under demand provides the following figures: 3.650 Euros/Kg. for the FC1 alloy and 3.950 Euros/Kg. for the FC2 alloy

From the cost analysis, it is derived that the main cost is, by far, the raw materials costs, accounting more than 90 % of the totals cost. Among the raw materials, primary aluminium accounts approximately half of the total material costs for both alloys and, therefore, its international pricing will have a big influence in the final price of the considered alloy selling price.

In both cases, nickel and zirconium pricing may have a significant contribution to the final selling price. In the case of nickel, primary metal has been considered for alloying, being its price fixed through international trade. In the case of zirconium, its addition is considered through the use of alloying tablets, where each tablet contains a precise weight of high purity alloying element, the balance being aluminium or a mixture of aluminium plus selected Na free, non-hydroscopic fluxes to accelerate dissolution and recovery. As a manufactured product, its final price is dependent on the formulation elements international pricing but also on the economies of scale.

In the case of alloy FC2 copper related cost should be mentioned because the relative amount of this alloying element in quite significant and the international pricing is relevant.