Comparison of different methods used to establish recrystallization conditions of gold alloys

1. Abstract

Recystallization affects both the structure and the mechanical properties of metals. The knowledge of recrystallization conditions for gold alloys is very important for optimizing the production process. Recrystallization conditions (temperature and time) depend on the type of alloy and the degree of deformation. The following methods for determination of recrystallization characteristics will be compared: hardness measurements, metallographic investigation and differential thermal analysis. Samples are heat treated in cycles with temperature and time as variable. We will take into account 14 ct yellow alloy. They are tested with different procedures to allow the comparison of methods for determination of recrystallization characteristics.

1. Introduction

Many gold articles are produced by means of mechanical operations such as drawing, rolling, pressing, etc.

If a metal undergoes plastic deformation at room temperature, its microstructure is greatly modified by the deformation and the metal is said to be work hardened.

If the metal is heated at increasing temperatures, a number of changes of varying degree take place until, at a certain temperature (over a certain duration) the work-hardened microstructure takes on a crystalline form, i.e. it is no longer deformed, and the metal's ductility and plasticity are restored.

The phase transformations of metals in the solid state are therefore fundamental to thermomechanical processing and likewise the ability of determining the applicable recrystallization conditions.

This paper aims to determine the possibility of an alternative and quick method to obtain correct values in a simple way for obtaining correct and reproducible values of a phenomenon of such fundamental importance to the productive cycle, taking as reference the traditional techniques (micro-structural analysis and micro-hardness) suitable to define annealing conditions.

After analyzing the various methods available, and following extensive bibliographical research of similar applications to other metal alloys, it was decided to intensify our research by concentrating on the use of differential thermal analysis (DTA).

It must be noted that thermal analysis instruments reproduce continuous annealing conditions, whereas in heat treatments for the analysis of hardness and grain size, specimens are subjected to batch annealing processes.

Thermal analysis offers many advantages, amongst which the rapidity in creating recrystallization conditions, the scientific reproducibility of the resulting data, as well as the small amount of material necessary for the test.

2. Theory

The plastic deformation of metals at room temperature mainly takes place as a result of the movement of pre-existing dislocations, together with the reproduction of new dislocations (via the Frank-Read mechanism).

A single crystal may become axially deformed by the action of slip of a single packet of parallel planes, whereas in polycrystalline materials, the different orientation of adjacent crystals favors slip along several intersecting planes. The overall result is that as the dislocations move, they end up obstructing each other.

Depending on whether the intersecting dislocations are edge or screw dislocations, a line of vacancies or climb may be created. Alternatively, part of a dislocation may be deviated onto a slip plane which is parallel to the original one (cross-climb). The overall effect is that the structure of a work-hardened metal is disturbed by an excess

of points defects and by an increase in dislocation density. Besides, one must also consider the effect of grain boundaries.

The dislocations that have moved pile up against the grain boundaries until the latter ultimately break. The microstructural outcome of this upheaval is that the crystals deform and elongate along the direction of the force applied, restoring anisotropy to the material, and finally break, increasing the surface area of grain boundaries.

The amount of cold-work carried out can be expressed by the degree of work hardening, defined as the percentage reduction of the deformed section:

$$i\% = \frac{S_0 - S_f}{S_0} \cdot 100$$

where S0 and Sf represent the cross-sectional area before and after cold-working respectively.

The mechanical properties of metals vary considerably with the degree of work hardening - the ultimate tensile strength, yield strength and hardness increase, whereas ductility, toughness and impact strength decrease.

Work hardening is also accompanied by important variations in the physical and chemical properties - density and electrical conductivity decrease slightly, magnetic permeability and corrosion resistance decrease considerably, whereas the coercive force, the coefficient of thermal expansion and the compressibility factor increase.

The crystalline condition of the material prior to deformation can be restored by subjecting the material to a heat cycle known as recrystallization annealing, and is governed by nucleation and grain growth processes.

During this treatment a number of transformations take place. These can be divided into three successive stages, as follows:
1.   Recovery
2.   Recrystallisation
3.   Grain growth

When we speak of recovery we are referring to the partial reinstatement (as a result of heating) of some of the material's properties without appreciable changes in the microstructure; these properties are then restored completely during successive heating at a higher temperature.

Since the annealing temperature which brings about recovery is relatively low, only the most mobile imperfections in the lattice structure are affected. The overall process consists of a simple microstructural rearrangement, which nevertheless has a considerable effect on the reduction of internal stresses, electrical resistance and material corrodibility; on the contrary it does not affect mechanical properties.

An important feature of the process is that at a certain temperature the process slows down with time; further, the degree of recovery obtainable over a certain time period increases as the temperature increases.

It is often difficult to define the dividing line between recovery and recrystallisation. This is because recovery plays an important role in the nucleation that takes place during recrystallization itself.

In order to restore the equilibrium of a work-hardened metal completely, both as regards the microstructure as well as its mechanical properties (which are not very sensitive to recovery), true and proper recrystallization is necessary: in this case the material must be provided with a higher thermal energy, i.e. one that is high enough to regenerate the structure completely. Such a process takes place by diffusion, via solid-state nucleation and grain growth mechanisms, and is a heat-activated process.

In any case, nucleation only occurs if the atoms possess the activation energy necessary to form permanent nuclei. Hence for a given annealing time there exists a minimum temperature at which recrystallization will occur. This minimum temperature varies with the degree of work hardening.

It is also worth mentioning that the properties of metals and alloys depend greatly on grain size, hence the heat treatment must be carried out as a function of the optimal size suited to the purpose.

The average grain size after annealing mainly depends on the degree of cold-work carried out on the material, on the annealing temperature and on the duration of the annealing process. By studying the way in which each independent variable influences grain size, the following practical/general considerations can be made:

i. the higher the degree of cold-work, the smaller the final grain size will be; in this case, in fact, the material contains several zones whose energy content is sufficiently high to let nucleation prevail over grain growth.
ii. below a certain degree of work hardening, deformation is slight and the driving force created is less than the necessary activating energy, hence annealing does not generate new crystals, but only favors recovery and grain growth of the work-hardened crystals;
iii. the final grain size increases as the temperature and annealing time increase;
iv. the higher the degree of cold-work, the lower the temperature will be at which recrystallization begins;
v.the longer the duration of the annealing process, the lower the temperature needed will be.

3. Experimental

An alloy having the composition shown in table 1, which we shall call ALLOY1, was used for our tests.

Table 1-Composition of the tested alloy

	Au	Ag	Cu	Zn
Alloy1	585	100	275	60

This is a very common yellow gold alloy. Alloy was produced with certified pure metals (> 99,99%) Physical proprieties of ALLOY1 are showed in table2:

Table 2-Fisical proprieties

	Density [g/cm3]	Tsolidus [°C]	T liquidus [°C]
Alloy1	12,99	839	854

4. Samples Preparation

After weighing, 200 grams (0,001 g accurancy) of ALLOY1 were melted in an protected atmosphere in an induction furnace with grafite crucible and poured into vertical ingot mould. The ingot obtained was rolled until 74% reduction was achieved and was then remelted in the same furnace. Another reduction to 74% was carried out, after which the plate was melted with under vacuum furnace with bottom pouring and protected atmosphere.In the end an 8 mm-thick ingot was obtained. The surfaces in contact with the ingot mould were cleaned by removing about 0.1 mm of material from both faces. Initial rolling with 50% reduction was carried out, yielding a plate thickness of 3.9 mm. 20 mm were then cut off the lower part of the ingot and about 60 mm from the upper part. The plate was then subjected to annealing at 650°C for 30 minutes in an electric furnace in a protected atmosphere.
At this point the material was subjected to a second rolling process with 74% reduction, achieving a thickness of 1 mm, followed by another heat treatment at 650°C for 30 minutes. A third and final rolling process with 74% reduction was carried out, yielding the final thickness of 0.26 mm. In each steps the plate was polished in both sides.
This procedure was actuated in order to assure maximum structural homogeneity in the resulting laminate.
The samples used for testing - precisely 139 medals measuring 16 mm long, 8 mm wide and 0.26 mm thick - were then cut from the thus obtained plate. The medals were numbered progressively so as to be able to examine the homogeneity of alloy composition both along the direction of rolling (and hence on different medals), as well as orthogonally to the direction of rolling (hence within the medal itself). Some medals were sent to external laboratories for analysis, to check the homogeneity of alloy composition between the various medals. Three different chemical analysis techniques were used: Cupellation (UNI EN ISO 11426:2000) for gold determination, ICP for Ag,Cu;Zn determination, XRF for all metal to detect the homogeneity . The following table3 lists the results obtained.

Table 3 – Analysis of the tested alloy

Position	Technics	Au	Ag	Cu	Zn
	ICP	-	-	-	-
Head	XRF	585,4	100,1	254,9	59,6
	Cupellation	585,3	-	-	-
	ICP	-	101,3	253	60,2
Pre-Middle	XRF	585,5	99,3	255,5	59,7
	Cupellation	585,4	-	-	-
	ICP	-	-	-	-
Middle	XRF	585,1	100,2	255,3	59,4
	Cupellation	585,4	-	-	-
	ICP	-	101	254	59,6
Post-Middle	XRF	585,4	99,3	255,5	59,8
	Cupellation	585,6	-	-	-
	ICP	-	-	-	-
End	XRF	585,5	99,1	255,6	59,8
	Cupellation	585,4	-	-	-

In view of the above results, the medals may be considered homogeneous and of constant composition.

5. Traditional Method

There are essentially two techniques presently used for determining and defining the right recrystallization conditions, by plotting the annealing temperature against annealing time and against the degree of cold-work (%). The two are: These are:
1. Hardness method: Analysis of the hardness resulting from heat treatment at different temperatures and for different durations at each temperature
2. Grain sizes method: Analysis of the average grain size following heat treatment at different temperatures and for different durations at each temperature.

For the above determination the medals were subjected to annealing at different temperatures for different durations. The following annealing

temperatures were chosen: 100,200,300,400, 500, 550, 575, 600, 625, 650, 675, 700, 750, 800 °C with the following annealing times: 2, 6, 18 minutes. Different medals were subjected to each of the above temperatures for each of the three different durations. These were mounted and polished and subjected to hardness testing after which they were chemically etched to carrying out average grain size.

Laboratory electrical furnace "Carbolite CWF1200" with protective atmosphere was used for preparation of samples.

4.1 Hardness Method

A Digital Microhardness Tester (Future_Tech FM-7e) was used.

We did five test for each sample; operating conditions were 200 grams and 15 seconds. The reported value is the average obtain (ASTM E384-99). The results are shown in table 4 that follows.

Table 4-Hardness sperimental points

Temperature [°C]	Time=2 min[HV 200/15]	Time=6 min[HV 200/15]	Time=18 min [HV 200/15]
25	302	302	302
100	296	300	301
200	309	313	316
300	310	304	305
400	301	286	292
450	268	270	240
500	243	206	171
550	193	170	154
575	172	157	149
600	172	157	153
625	162	152	148
650	153	151	145
675	156	148	150
700	155	149	145
750	151	146	144
800	143	141	142

Hardness trend is shown in figure 1:
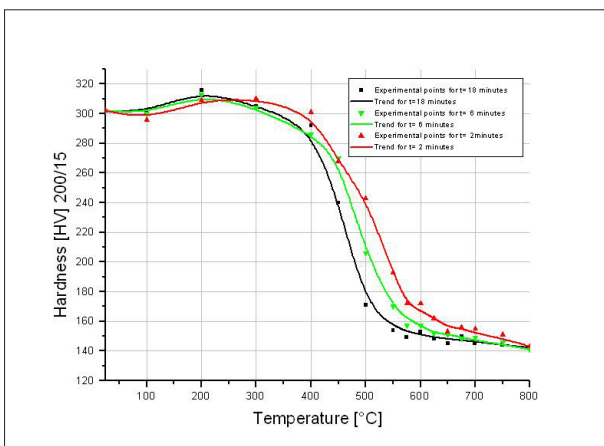
Figure 1-Hardness trend

Annealing ranges of temperatures can be defined by the analysis of the hardness values obtained at three different time conditions.

This method presents precision limits due to the flattening of values in high temperatures.

The results are reported on the table 5

Table 5- Annealing Temperatures range obtained by Hardness method

Time [minutes]	Annealing temperatures [°C]
2	> 750
6	650-675
18	625-650

4.2 Grain sizes Method

A metallurgical Microscope (Nikon Eclipse Mod. ME600) with Nikon digital camera DXM1200 was used.

We did three tests for each samples. The reported value is the average obtained (ASTM E112-96). The results are shown in Table 6.

Table 6-Grain sizes experimental points

Temperature [°C]	Time=2 min[µm]	Time=6 min[µm]	Time=18 min[µm]
575	Not formed	Not formed	14,9
600	Not formed	Not formed	15,5
625	Not formed	15	28,8
650	Not formed	15,2	28,6
675	11,4	21,6	35,1
700	13,9	27,2	47,5
750	16,5	33,7	65,4
800	27,6	67,1	133,3

Grain sizes trend are shown in figure 2 below.
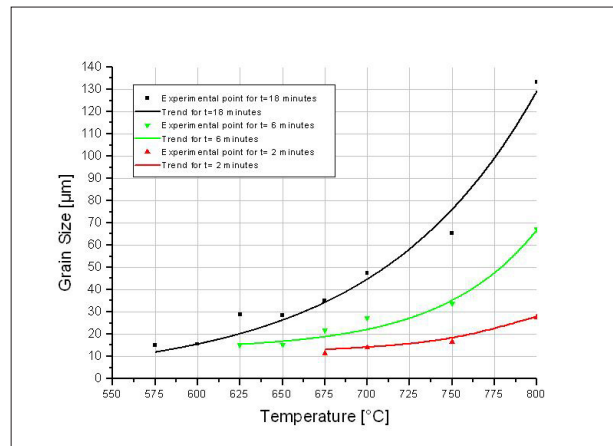
Figure 2-Grain sizes trends

Microstructure pictures are reported in Appendix A

Annealing ranges of temperatures can be defined by the analysis of the grain sizes values obtained at three different time conditions.

This method presents high precision because it shows clearly when grain is formed and when grain growth. The results are reported in Table 7

Table 7- Annealing Temperatures range determinated by Grain sizes method

Time [minutes]	Annealing temperatures [°C]
2	> 750
6	675-700
18	625-650

With results of two methods, it is possible to have the ideal annealing conditions of the ALLOY1 to be used as standard reference.

The grain size values (confirmed by the hardness values) of the samples annealed for 18 minutes show the ideal annealing temperature to lie between 625°C and 650°C, since the average grain size measured between these two temperatures is almost constant (about 26-30 microns). On the contrary, below and above this temperature range, the differences in grain size are high.

Hence the reference values for annealing temperature and time were taken to be 637 °C (± 10) and 18 minutes respectively. This temperature (Trf18) will be used as reference to compare with DTA method

4.3 Differential Thermal Analysis Method

We choosed DTA method after preliminary investigation, because we detected an accurate and relevant phenomenon in the range of temperature 550°C - 750°C.

DTA is a technique in which the change of the difference in temperature between the sample and a reference sample is analysed while they are subjected to a temperature alteration.

Before deciding on the type of instrumentation to be used, a number of preliminary tests were also carried out using DSC (Differential Scanning Calorimetry).

Furthermore, white gold alloys (which have higher annealing temperatures with respect to those of yellow gold) will be studied at a later stage using the same technique.

The TG/DTA instrument, after suitable calibration, offers a higher resolution and quicker signal response at the temperature range being observed (600-750°C). The instrument used was a "TG/DTA 6300" (Seiko Instruments Inc.) and instrument data is shown here in Table 8:

Table 8-Features of the TG/DTA 6300 used

	TG/DTA 6300
Weight MeasurementSample Weight	Horizontal differential balance methodMax. 200 mg
Temperature Range	Room Temperature to 1500 °C(normal 1300 °C)
Heating RateTG Measurement RangeDTA Measurement RangeDTG Measurement Range	0.01 °C/min ~ 100.00 °C/min ±0.1 mg ~ ±200 mg ±2,5 µV ~ ±2500µV 0.5 mg/min ~ 1 g/min
Gas Flow	Max. 1000 ml/min

Temperature calibration was carried out by inserting the melting points of four certified metals, precisely indium, zinc, aluminum and silver.

It is hereby emphasized that the whole system was calibrated on the melting point of aluminum (660 °C), this being considered the temperature closest to the recrystallization temperature expected for the alloy under examination.

Before proceeding with final experimentation, tests were carried out to establish the optimum testing conditions, by studying the influence of:

a. The type and flow rate of the protective gas
b. The heating rate (°C/min)
c. The mass sample weight
d. Oxidation of specimens
e. Crucibles type

a. Type and flow rate of the protective gas
During the first blank reference tests (with annealed specimens) a mixture of 95% Nitrogen and 5% Hydrogen was used, since this mixture lends itself well to tests for the determination of melting points. At around 560°C a phenomenon was observed that was attributed to the presence of air in the heating chamber and to oxidation of the sample.

When pure Argon (>99.999) was used this phenomenon did not occur, hence argon was chosen to be the protective gas.

Before feeding the gas continuously, the chamber was subjected to a vacuum and washed with gas, each operation being carried out three times.

The gas flow rate was 50 ml/minute for all the tests, kept constant throughout the entire test operations.

b. Heating rate (°C/min)
Preliminary testing was carried out using the following heating rates: 5, 7, 10, 20 and 30 °C/minute.

Amongst these, the best results, under several aspects, were yielded by the heating rate of 10°C/minute.

c. Mass sample weight

Preliminary testing was carried out at a rate of 10°C/minute on various sample weights - 21, 42 and 84 mg. Since no significant differences were noticed in the results, it was decided to effect all testing with a mass sample weight of 42 (±0.5) mg, given that this corresponds to the weight of a single disc specimen.

d. Oxidation of specimens

Notwithstanding the use of a protective gas, the specimens were seen to undergo a small degree of oxidation whereby it became necessary to investigate how this oxidation affected test results (see figure 5).

This was done by testing a single specimen first in the work-hardened state and then, without opening the heating chamber of the instrument, by testing it twice in the annealed state.

The curves yielded by the two tests of the annealed specimen were the same, whereby it was concluded that although some degree of oxidation would occur, this would not affect test results.

In confirmation of the above, another specimen that had been subjected to two annealing cycles in a batch furnace at 750°C for 30 minutes, was tested. The same results were obtained.

e. Crucibles type

Both platinum and pure alumina (Al2O3) crucibles were considered, and since, as expected, the signal sensitivity yielded by the platinum crucibles was no greater than that of the alumina crucibles, the latter were chosen for practicality's sake.

Final experimentation proceeded by carrying out various tests under the following conditions:

Protective gas type		Argon (99.999)
Gas flow rate	ml/m	50
Mass sample weight	mg	42 (± 0.5)
Heating rate	°C/min	10
Crucibles type		Alumina

The work-hardened specimens used consisted of 3.85 mm diameter discs (a size which allows the surface of the TG/DTA crucible to be covered completely), obtained by shearing the medals.

The annealed specimens used for reference purposes were obtained by annealing the above discs at a temperature of 750°C for 30 minutes.

The two alumina crucibles weighed the same (about 88 mg) and the sample to be analyzed was always positioned in the crucible on the right hand side.

A number of reproducible curves at 10 °C/min heating rate were obtained wherein the phenomenon of recrystallization could be detected as shown by the following (figure3).
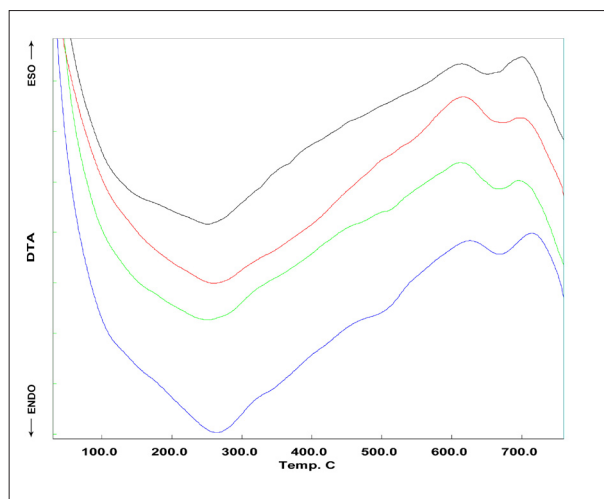
Figure 3-Curve DTA obtained at heating rate of 10 °C/min

Three points in particular may be determined which we shall call Ts, Tmax and Te (see figure 4), where:

Ts	may be considered to be the temperature at which recrystallization begins
Tmax	is the temperature at which the instrument detects the highest recrystallization rate
Te	may be considered to be the temperature at which recrystallization ends

The data obtained are reported on Table 9:

Table 9-DTA data obtained with heating rate of 10°C/min

	Ts[°C]	Tmax[°C]	Te[°C]
Test 1	619,5	652,3	718,8
Test 2	613,2	635	701,3
Test 3	614,1	636,5	706,2
Test 4	617,2	642,2	698,8
Average	616,0	641,5	706,3
DVS	2,9	7,8	8,9

In some of the tests the signal will be noticed to peak slightly in the region of 510 °C. This peak may be assumed to be related to the phenomenon of recovery. The energy necessary for recovery to occur is very low, whereby it is possible that the instrument is not sensitive enough to detect this phenomenon each time. For this reason, and seeing that it is of no practical concern, this occurrence was not analyzed further but has simply been mentioned.
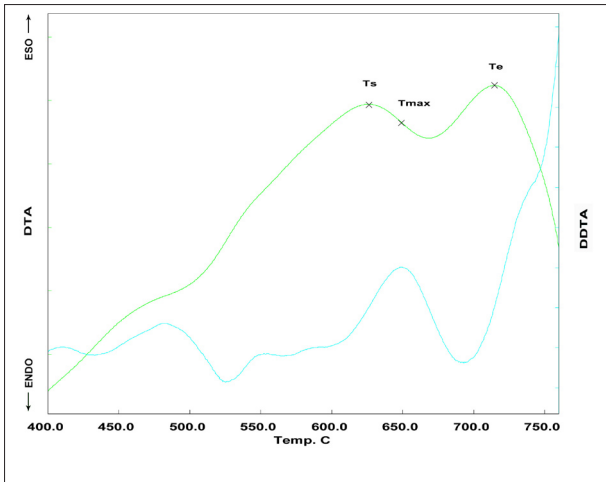
Figure 4-Ts, Tmax, Te points in DTA curve

In confirmation of the above it will be noticed that once the Te value is exceeded, the curve behaves as for an annealed specimen. The curve of the annealed specimen that was tested twice to examine the effect of oxidation on the resultant curve, as already explained before, is also shown in the following Figure 5.
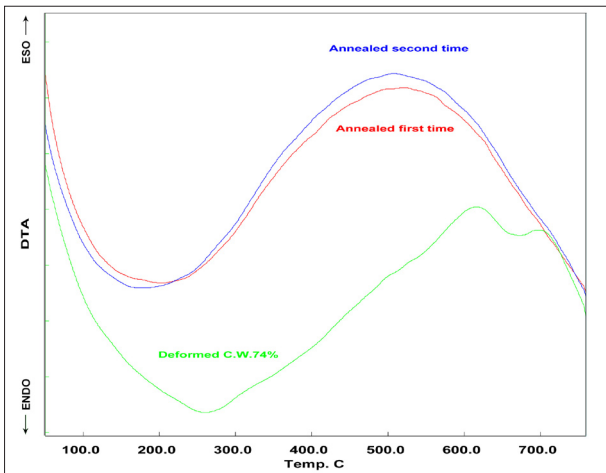
Figure 5-Cold-worked and Annealed DTA samples comparation

In figure 6 and Table 10 the Ts, Tmax and Te values were measured by the DTA for different heating rates.
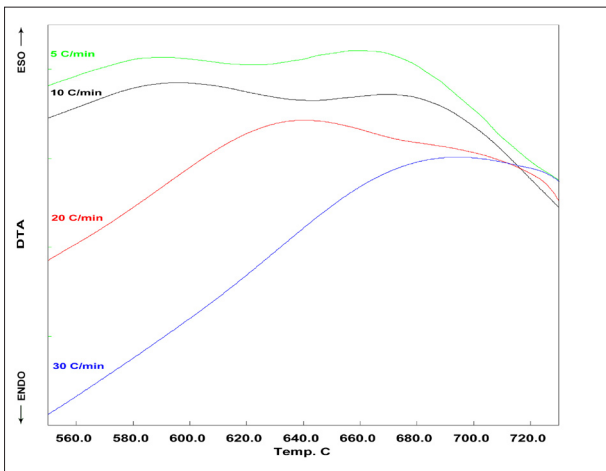
Figure 6-Trend DTA curves for different heating rate

Table 10-Ts, Tmax, Te value for different heating rate

Rate Heating [°C/min]	Mass [mg]	T start[°C]	T max[°C]	T end[°C]
5	85	583,6	598,1	668,1
5	85	585,4	608,5	669,3
5	42	585,6	609,2	665,5
5	42	586,2	607,4	668,1
7	85	602,9	616,1	687,2
7	85	604,1	630,7	694,9
7	42	596,5	627,1	690,6
7	42	595,4	637,8	688,1
10	85	626,2	653,1	719,7
10	85	617,2	652,6	711,9
10	42	619,5	652,3	718,8
10	42	614,3	636,5	706,2
20	85	652,2	675,1	740,1
20	85	657,6	685,1	738,4
20	42	647,1	663,8	723,8
20	42	648,1	677,3	742,2
30	85	Not detected	Not detected	Not detected
30	85	Not detected	Not detected	Not detected
30	42	Not detected	Not detected	Not detected
30	42	Not detected	Not detected	Not detected

In order to verify the results yielded by the analytical instrument, as well as to confirm the theories regarding Ts, Tmax, and Te, the DTA was simulated in the laboratory.

For this purpose disc specimens of cold-worked material were heated at the heating rate set at 10 °C/min and samples representing continuous annealing between 25 °C and 800°C were obtained at the following temperatures: 25, 160, 300, 400, 500, 550, 575, 600, 625, 650, 675, 700, 725, 750 and 800 °C. This simulates the conditions taking place in the DTA, the only difference being that at the temperatures indicated above, the samples were removed and quenched in water.

A laboratory electrical furnace "Carbolite CWF1200" with protective atmosphere was used for samples preparation.

These were mounted, polished and subjected to hardness testing, after which they were chemically etched to carrying out average grain sizes.

The results of "DTA simulation" hardness are showen Table 11:

Table 11-"DTA simulation" hardness experimental points

Temperature[°C]	Hardness[HV 200/15]
25	302
160	268
300	288
400	277
500	237
550	184
575	168
600	164
625	156
650	160
675	158
700	157
725	165
750	160
800	155

"DTA simulation" hardness trend is shown in figure 7:
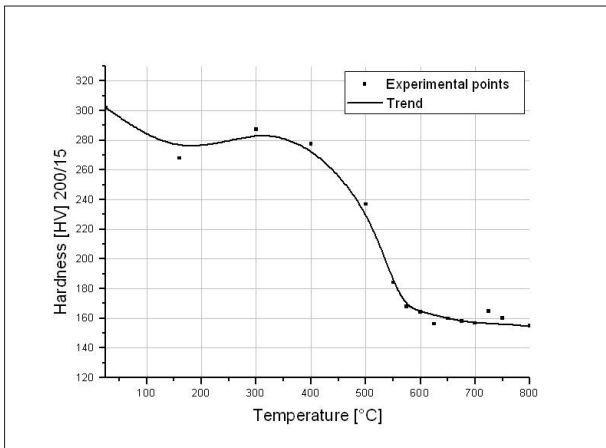
Figure 7-Trend of hardness in "continuos heating" for "DTA simulation"

The results of "DTA simulation" grain sizes are shown table 12.

Table 12-"DTA simulation" grain size experimental points

Temperature[°C]	Grain Sizes[μm]
575	Not formed
600	Not formed
625	14,7
650	14,8
675	18,1
700	30,5
725	45,5
750	53,7
800	90,9
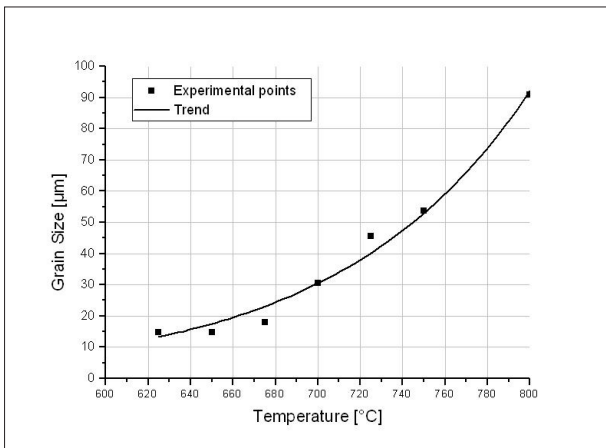
Figure 8-Trend of grain sizes in "continuos heating" for "DTA simulation"

6. Discussion of results

We did comparison between results of method DTA and DTA simulation (see figure 9 below)
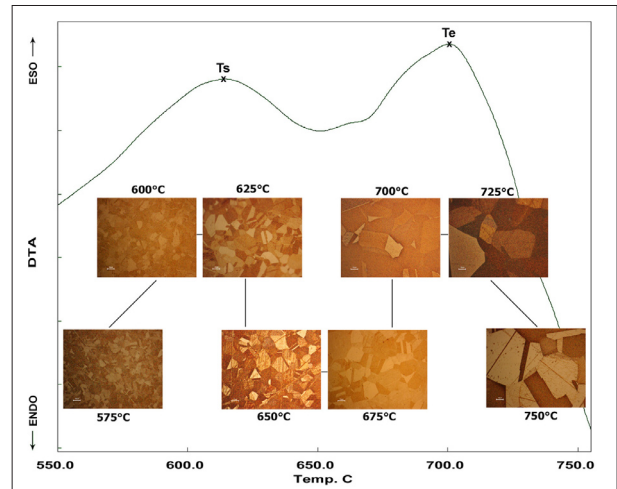
Figure 9-Comparison between DTA curve and DTA simulation photos

Examination of the metallographic plates used to determine the grain size shows that no grain crystals form before the temperature Ts is reached i.e., before recrystallization begins. Grain crystals are only observed above Ts and at first glance they also appear to be rather large. Between Ts and Te grain growth is seen to be limited (from about 26 microns to 30 microns). Above Te the grain size grows exponentially, which is why we consider Te to be the reference temperature at which annealing is complete.

To verify this we compared grain size in DTA simulation with grain size measured in traditional method with 18 minutes of annealing time (see Figures 10, 11 and 12).
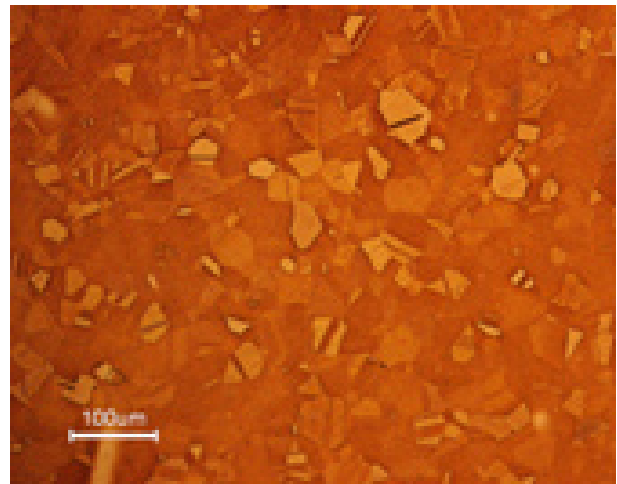
Figure10- DTA simulation 700°C
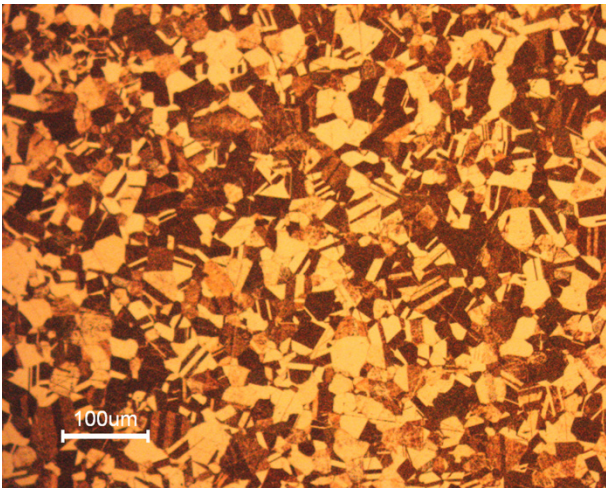
Figure11- Trad.method (625°C/ 18')

Figure12- Trad.method (650°C/ 18')

Conclusion of this comparison is that at Te temperature, the grain size measured in DTA simulation correspond with grain size measured in traditional method in the range temperature 625°C-650 °C (Trf/18).

Microstructure pictures DTA Simulation are reported in Appendix B. The data obtained at the different heating rates (refer to table 10 for different heating rates) show all the Te values to be higher than the Trf/18 temperature. Moreover the measured temperatures are proportional to the heating rate and are therefore higher for higher heating rates. This is because, for a given reached temperature, more energy is supplied at lower heating rates (longer annealing times).

It then follows that if the DTA is to be used for determining a fixed temperature and time for annealing in a batch furnace, the equivalent heating rate must first be determined. This means that the Te value of the DTA test must be matched to the Trf/18 temperature value for annealing in a batch furnace, after which the heating rate, which yields the same average grain size, must be found. The heating rate for which Trf/18 and Te are equal may be determined by extrapolating the graphs of Te at different heating rates
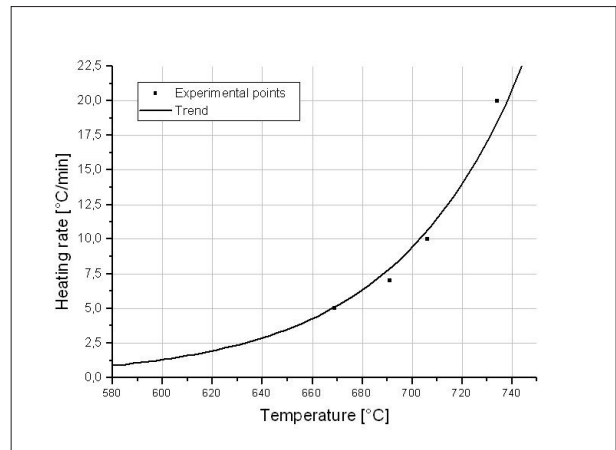
Figure 10-Trend Te at different heating rates

The heating rate, determined with this instrument, calibrated as specified and in the operating conditions described above, was found to be 2.5 °C/min.To verify the above, two DTA's were carried out at a heating rate of 2.5 °C/min.
Results of the DTA's are shown in figure 14 and in Table 13.
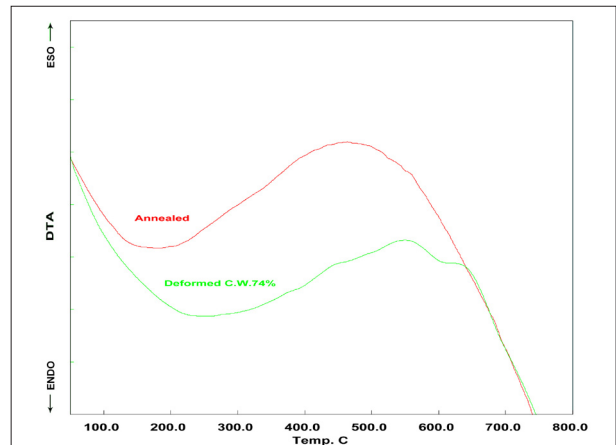
Figure 11-DTA curve at heating rate 2,5 °C/min

Table 13-DTA results obtained at 2,5°C/min heating rate

	Ts[°C]	Tmax[°C]	Te[°C]
Test1	557,6	585,3	634,3
Test 2	553,1	598,8	638,2
Average	555,4	592,1	**636,3**
DVS	3,2	9,6	2,76

This results confirm all our hypothesis.
To find the right recrystallization temperature of an alloy (with industrial condition batch furnace for 18 minutes), our DTA test must be performed with 2,5°C/min heating rate. This hypothesis must be confirmed with several future tests.

7. Conclusions

The main aim of this investigation was to compare two traditional methods, namely a microstructural test of cristalline grain and micro-hardness, which are used to define annealing conditions by means of a new method called differential thermal analysis (DTA).

Before going ahead with the comparison, it was necessary to define:

- whether it would be possible to observe a recrystallization phenomena using DTA.
- whether the said phenomenon could be reproduce and considered reliable.
- whether it could be used and compared with traditional methods to determine alloy annealing conditions.

The results obtain are summarized as follows:

a. Of the traditional test, the microstructural one was the most reliable. Microhardness furthermore confirms the results of microstructural tests. The comparison was made on this basis.

b. Recrystallization is unmistakably observed in the DTA curves.

c. Highly repeatable results were reached. However it must be taken into consideration that it took a long time to set up the instrument and especially define the ideal conditions to operate the experiment.

d. In line with above results we may consider that DTA represents an excellent method to define annealing temperature with reference to time. Once the relationship is indeed established between data using a traditional method and data obtained from DTA it is possible to define annealing conditions.

e. Whether DTA method can be applied for alloy characterization as yet has not been verified.

f. The system elaborated for static annealing conditions can be furthermore improved with more data.

In the future we would like to improve our understanding of:

- Verify the extend to which DTA can be applied to characterize alloys carrying out experiments on different carat, composition and colour alloys.
- The more data we will have based on experiments carried out on several alloys, the more we will determine the relationship between static annealing condition and DTA.

8. Acknowledgements

9. References

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Appendix A
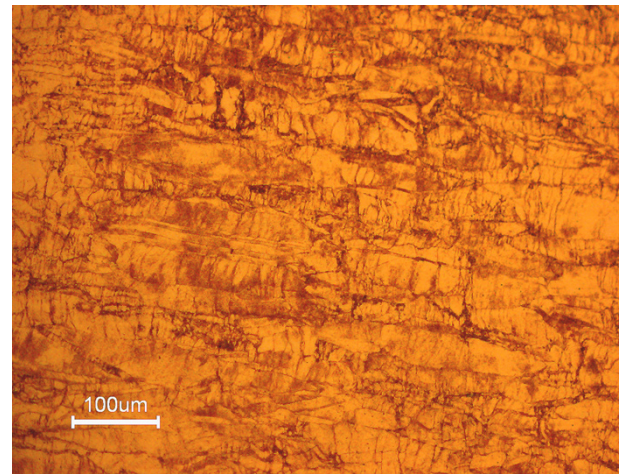
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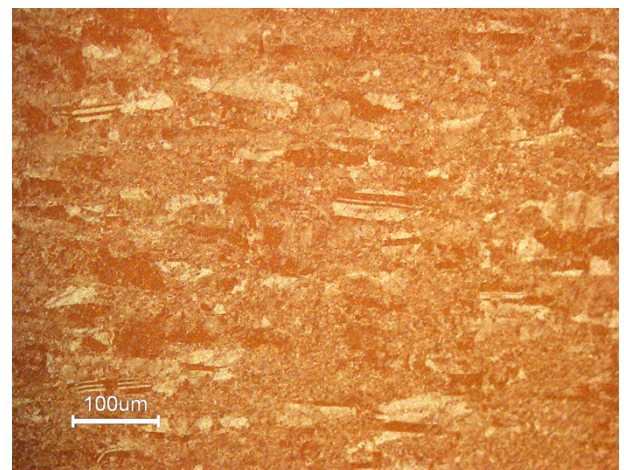
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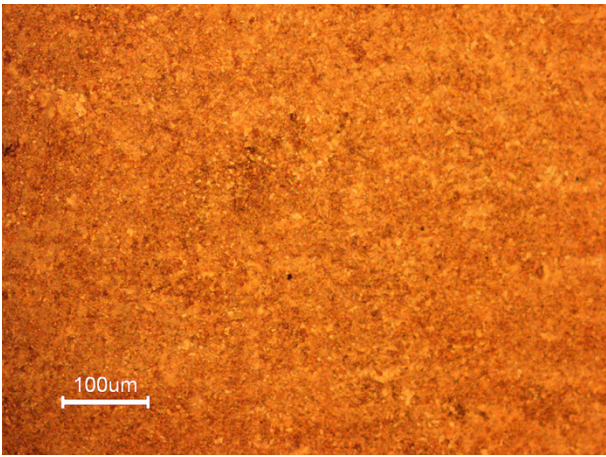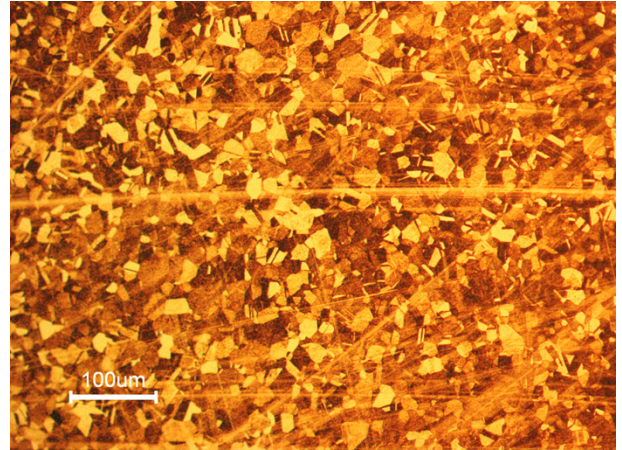
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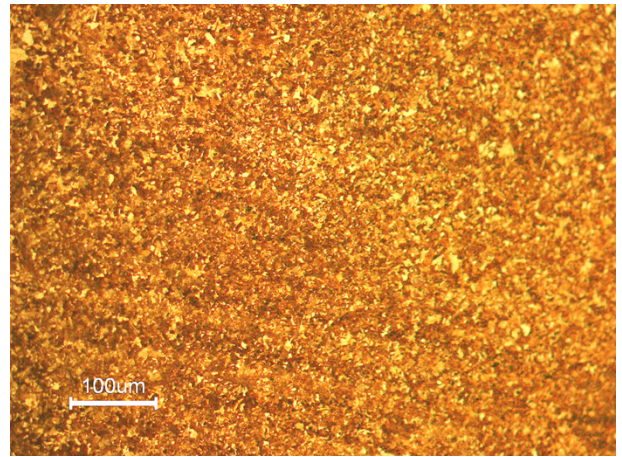
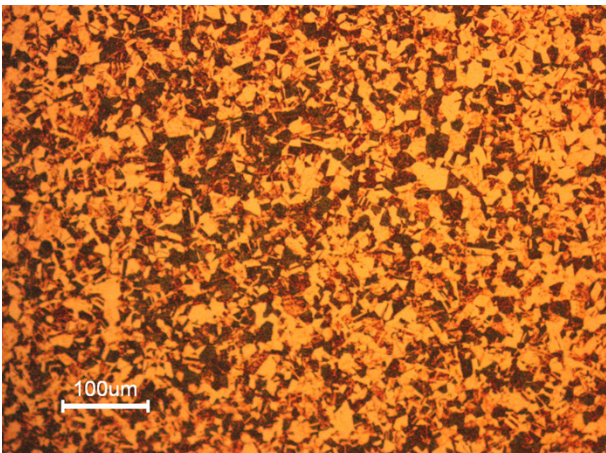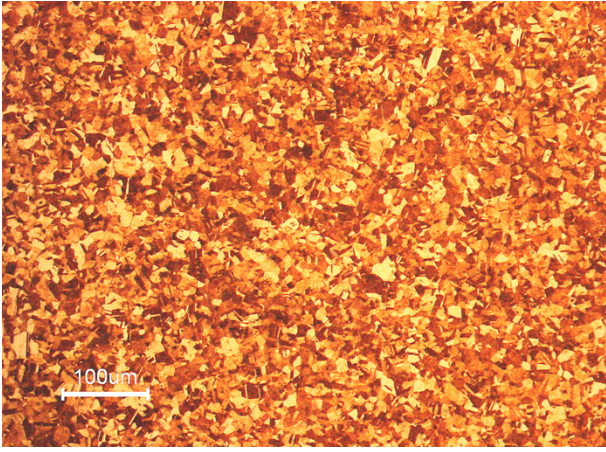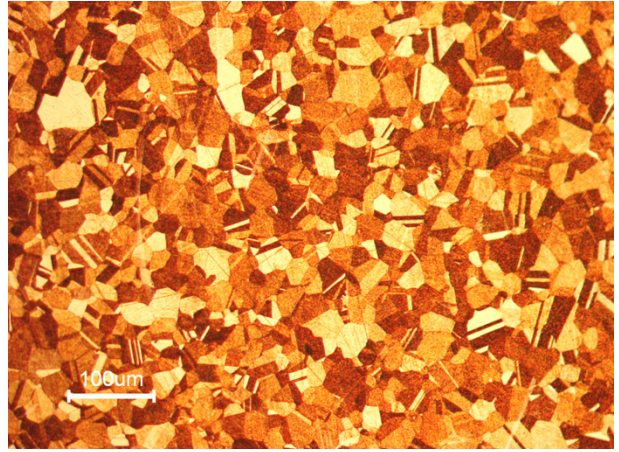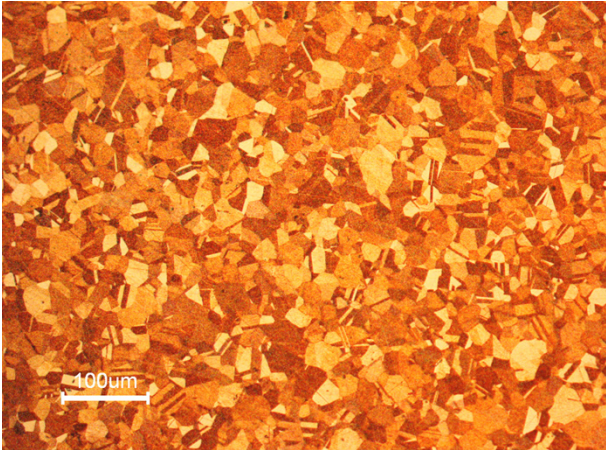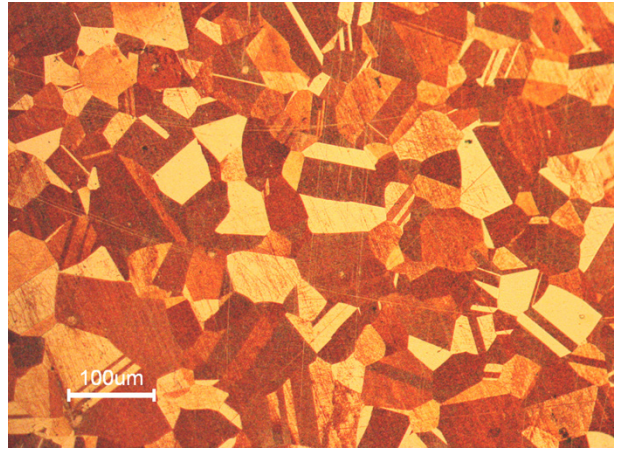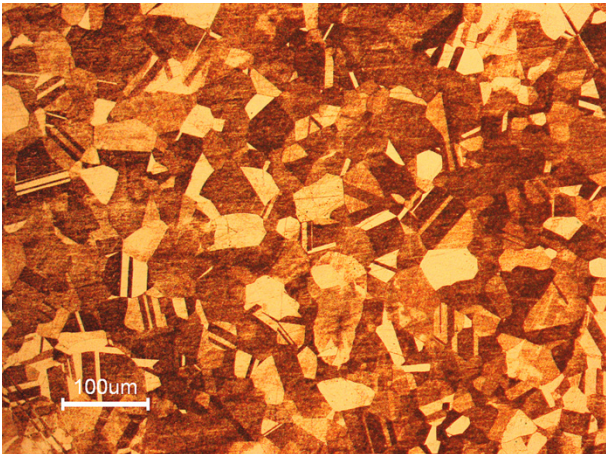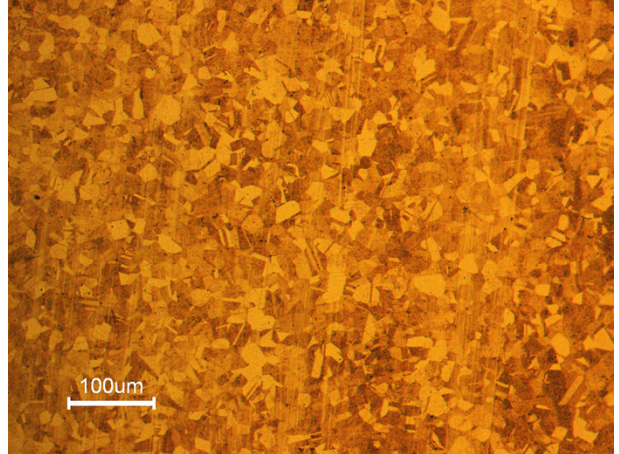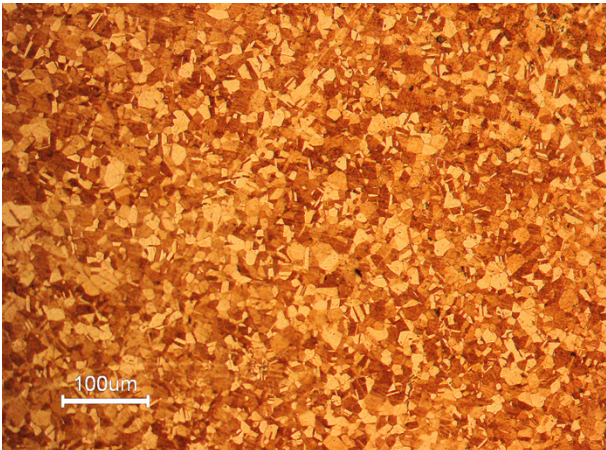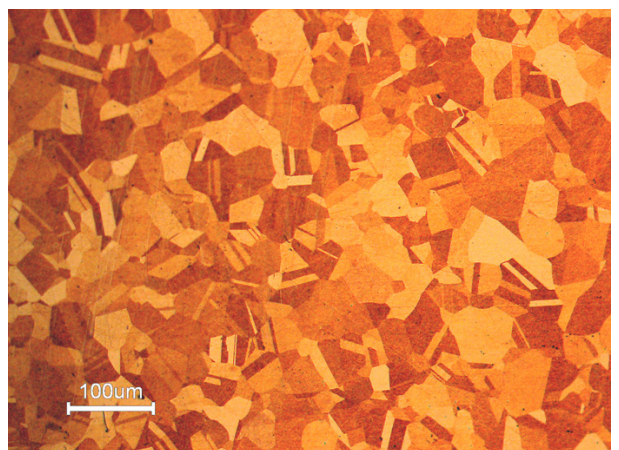**Traditional method: T=575°C Time=2min.**
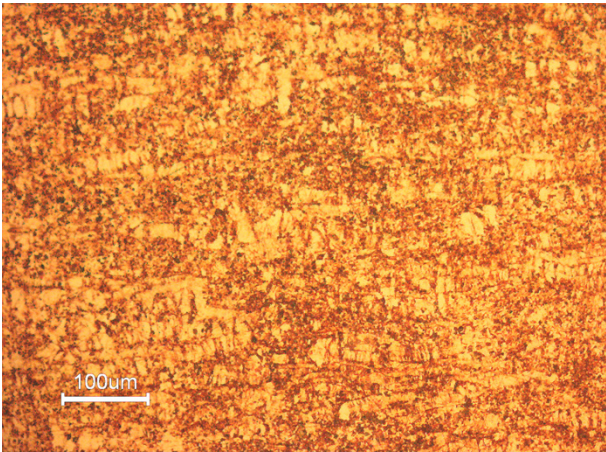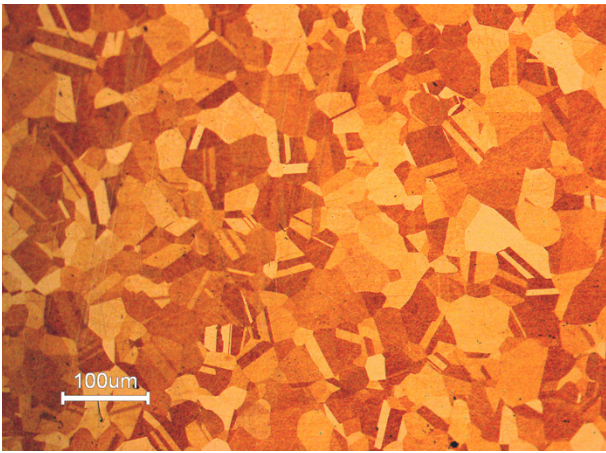

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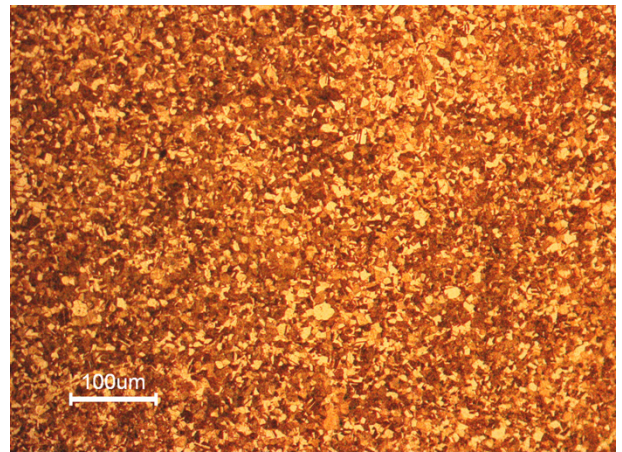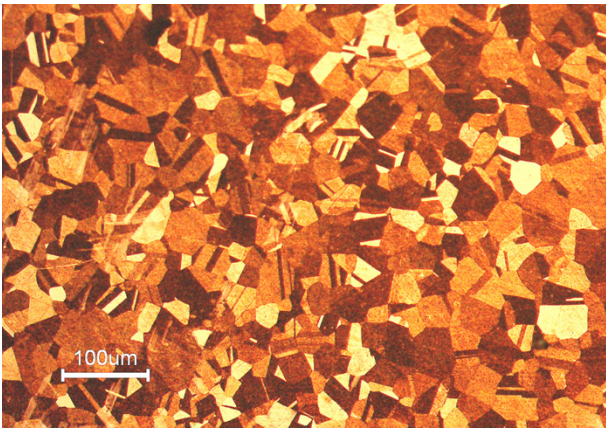

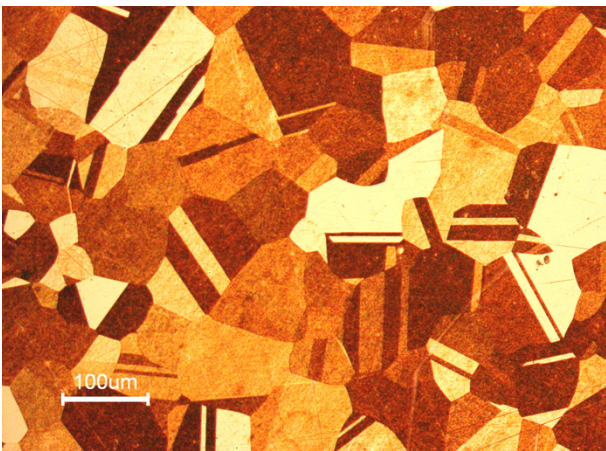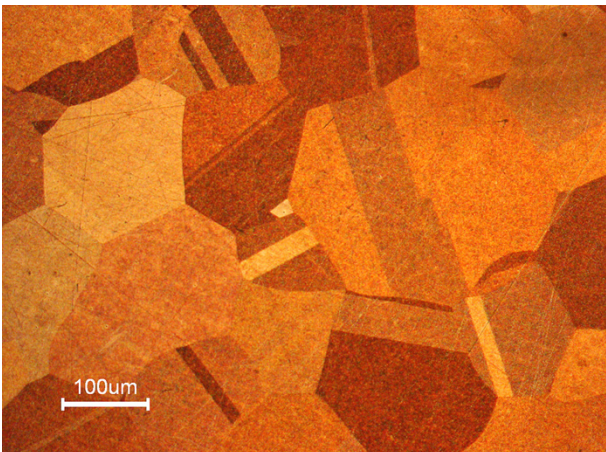**Traditional method: T=575°C Time=6min.**
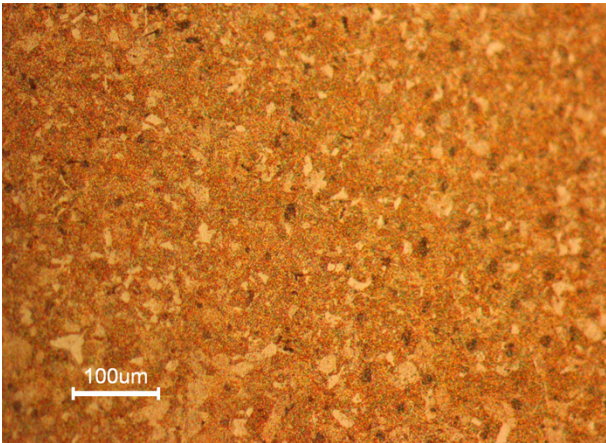
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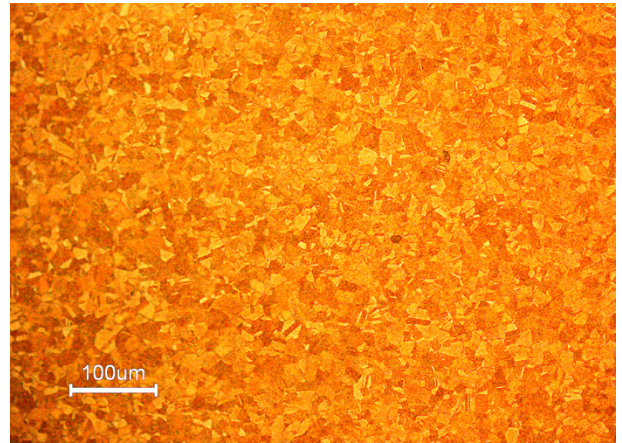
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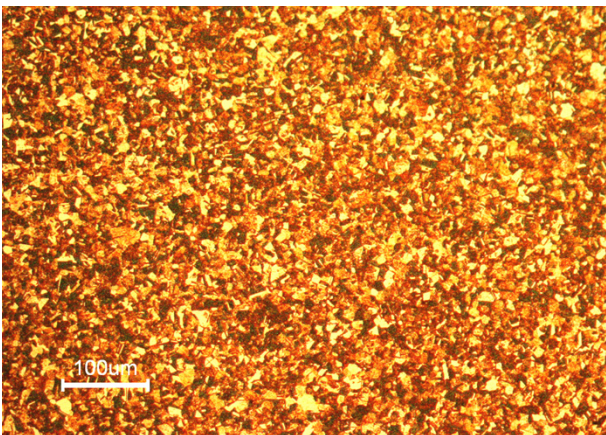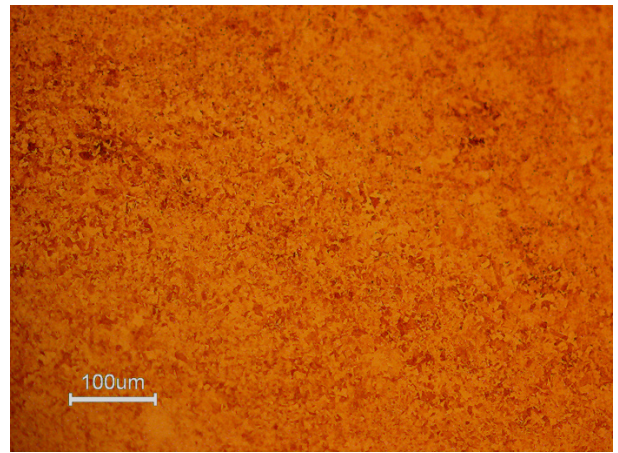

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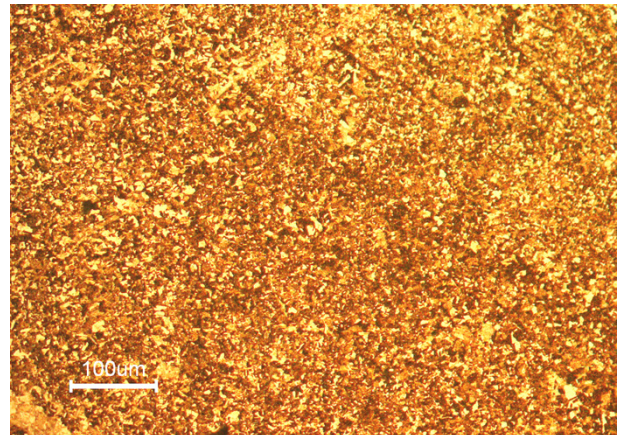
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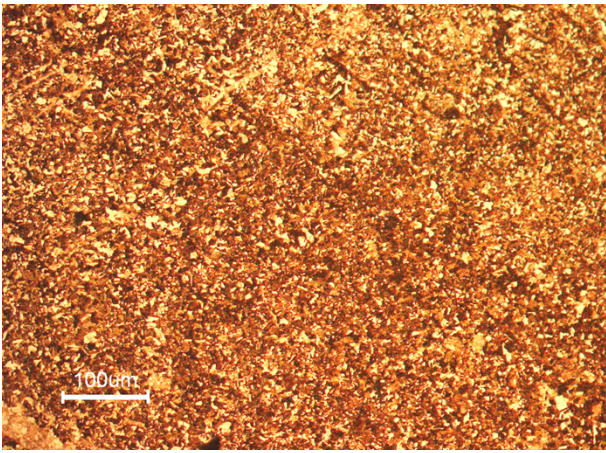
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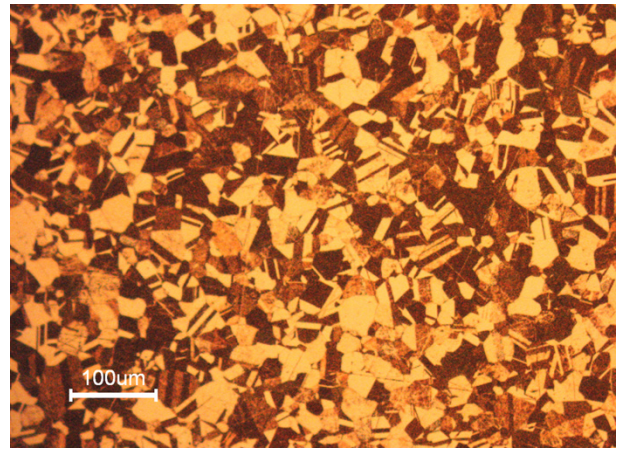
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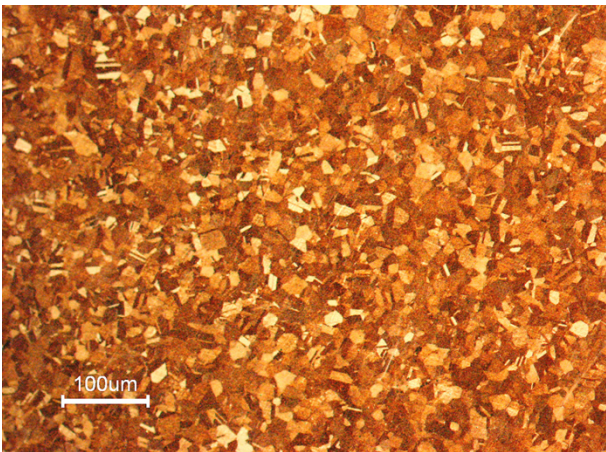
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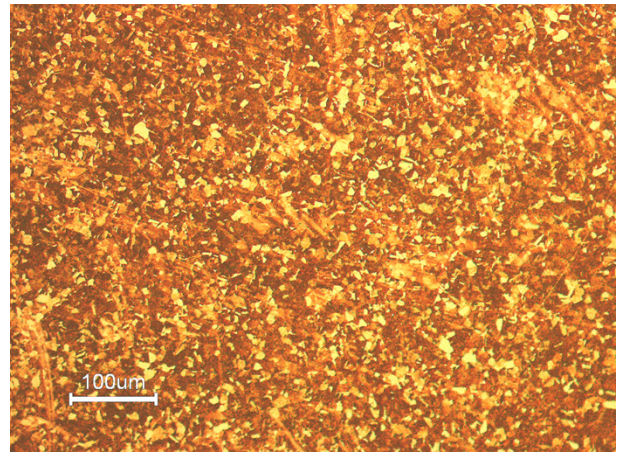
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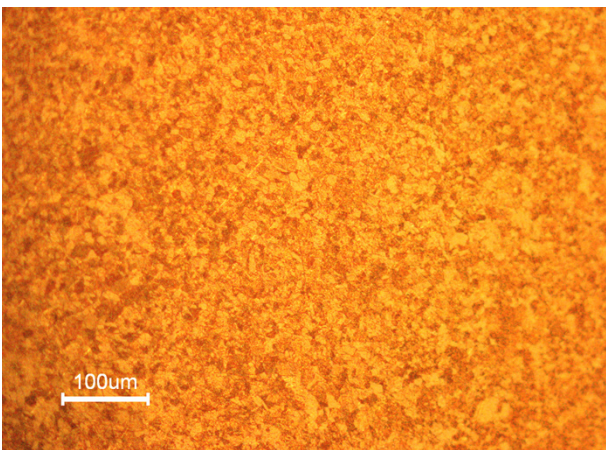
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Traditional method: T=650°C Time=6min.

Traditional method: T=625°C Time=6min.

Traditional method:T=650°C Time=18min.

Traditional method: T=675°C Time=2min.

Traditional method: T=700°C Time=6min.

Traditional method: T=675°C Time=6min.
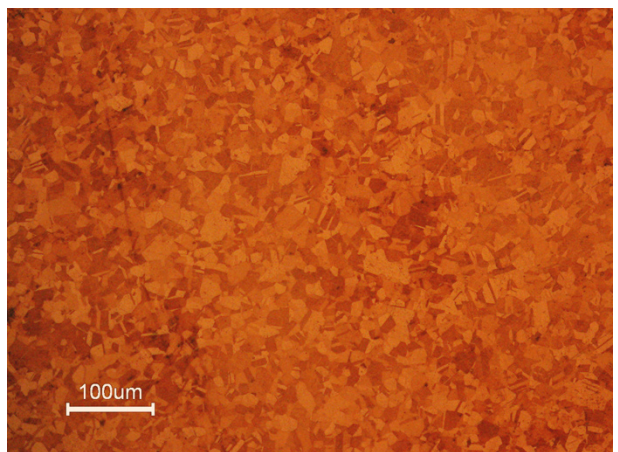

Traditional method:T=700°C Time=18min.

Traditional method:T=675°C Time=18min.

Traditional method: T=750°C Time=2min.

Traditional method: T=700°C Time=2min.

Traditional method: T=750°C Time=6min.

Traditional method:T=750°C Time=18min.

550 °C - DTA simulation

Traditional method: T=800°C Time=2min.

575 °C - DTA simulation

Traditional method: T=800°C Time=6min.
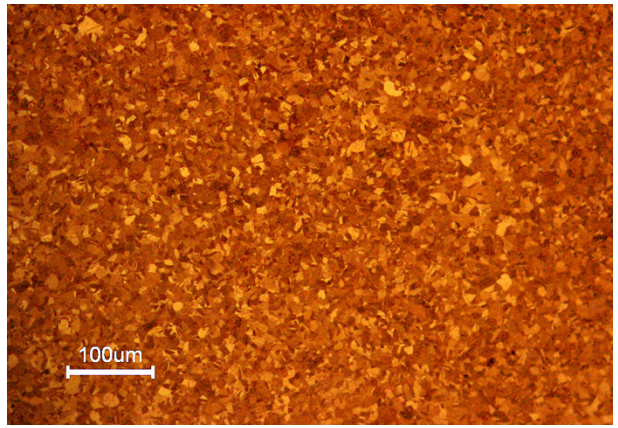
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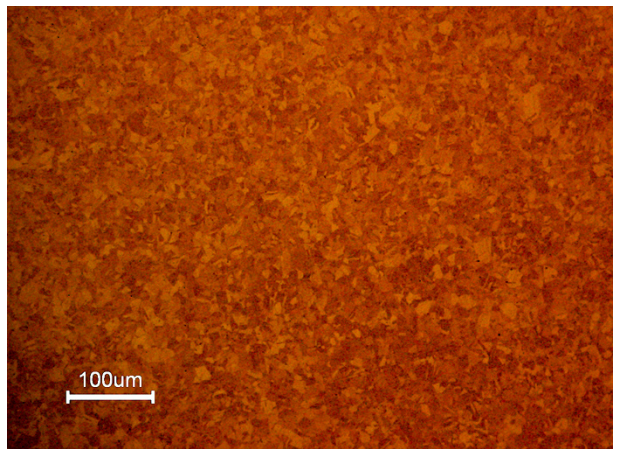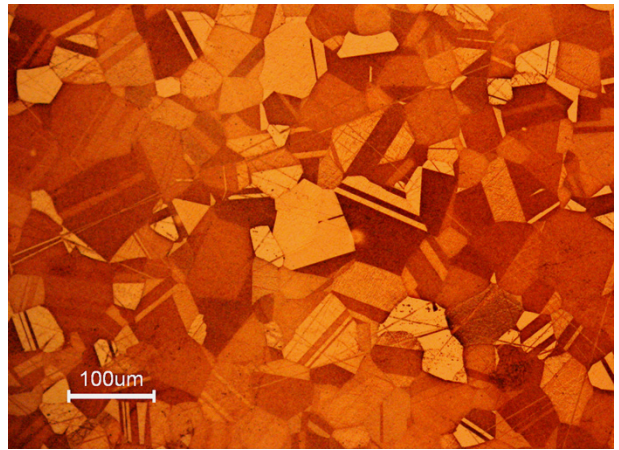

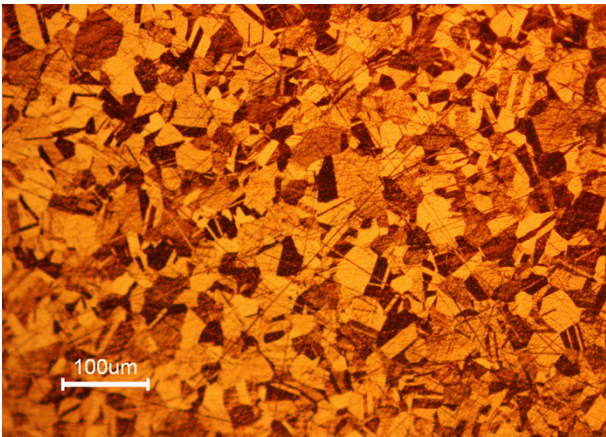**Traditional method:T=800°C Time=18min.**

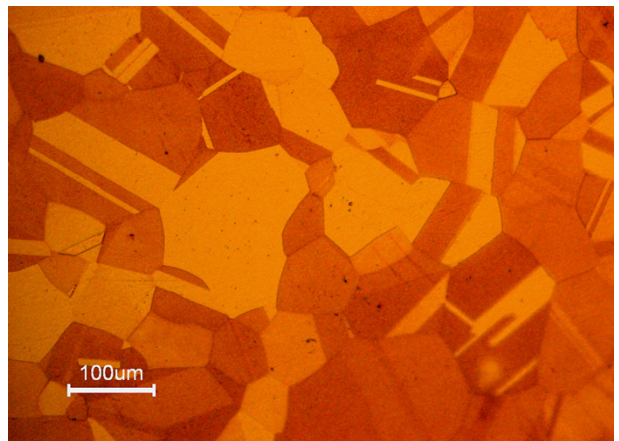**625 °C - DTA simulation**

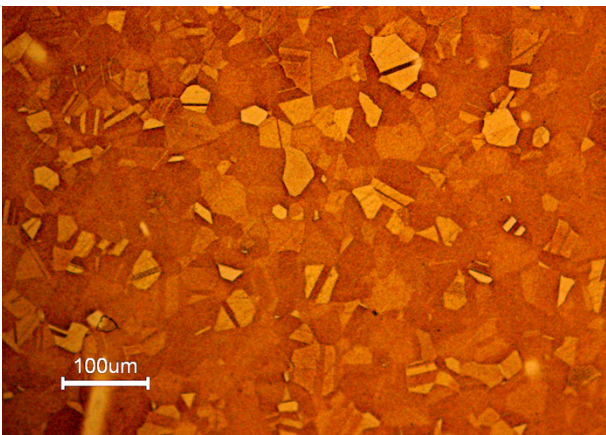**650 °C - DTA simulation**

750 °C - DTA simulation

675 °C - DTA simulation

800 °C - DTA simulation

700 °C - DTA simulation

725 °C - DTA simulation