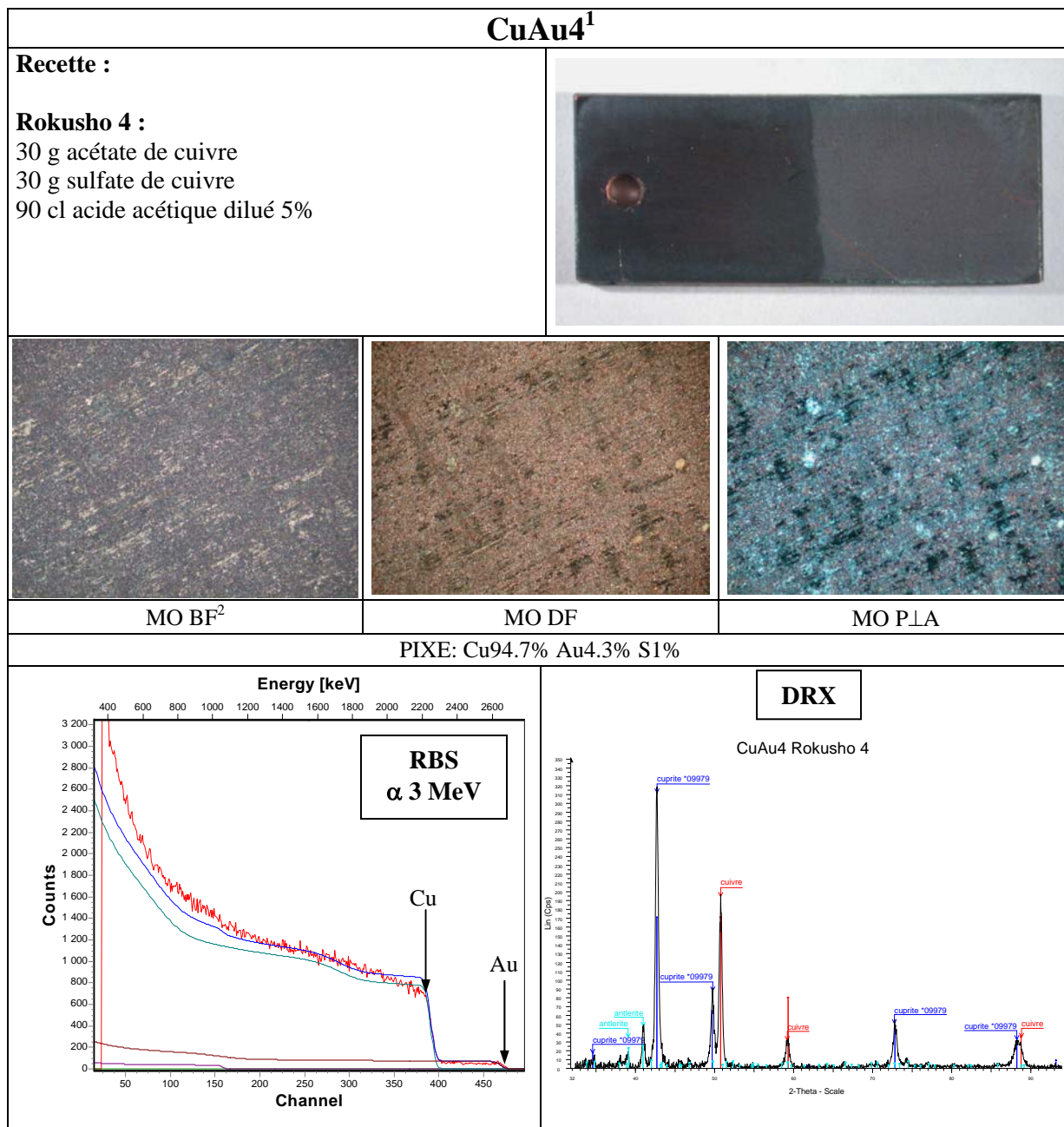


**Annexe 7**

**Les patines expérimentales obtenues par  
utilisation des recettes japonaises**

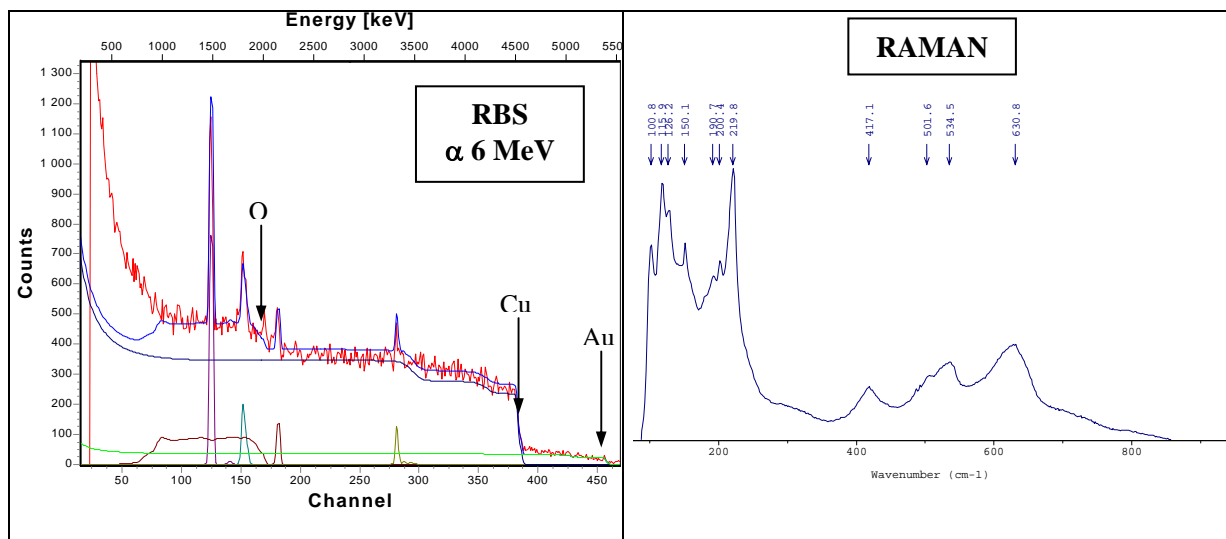
**Résultats d'analyses**





<sup>1</sup> La moitié des échantillons est vernie pour mettre en valeur la couleur de la couche de surface obtenue. Les analyses sont effectuées sur la partie non vernie.

<sup>2</sup> Le sigle MO signifie Microscopie Optique. BF indique que l'observation a été faite en fond clair (réflexion directe), DF qu'elle a été réalisée en fond noir (illumination oblique) et P⊥A qu'elle a été effectuée en lumière polarisée (polarisants croisés)



### Description

Composition Couche			Diffraction X																																																																
Cu40 O25 Au0.5 Porosité +rugosité Cu/Au=80	7000.10 <sup>15</sup> At/cm <sup>2</sup>		Cuprite décalée *0.9979																																																																
Cu66 Au1 O33 Cu/Au=66	9000.10 <sup>15</sup> At/cm <sup>2</sup>		Cuivre non décalé																																																																
Cu98.4 Au1.3 Cu/Au=75.7	Substrat		<b>Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes</b>																																																																
<b>Analyse XPS:</b> Or métallique détecté : Signal Au 4f7/2 détecté à 84,45 eV soit 0,45 eV de plus que la valeur théorique de Au <sup>0</sup> 4f 7/2 (Au <sup>3+</sup> 4f 7/2 : 86 eV)			<b>CuAu4 R4 - cm<sup>-1</sup></b>																																																																
			<table border="1"> <thead> <tr> <th>sommet</th> <th>Amplitude</th> <th>largeur</th> <th>Surface</th> </tr> </thead> <tbody> <tr><td>100,51</td><td>4704,43</td><td>6,17</td><td>22233,9</td></tr> <tr><td>11,65</td><td>8416,24</td><td>13,29</td><td>102392</td></tr> <tr><td>128,04</td><td>5267,3</td><td>8,22</td><td>40656,7</td></tr> <tr><td>150,3</td><td>4476,77</td><td>7,95</td><td>33774</td></tr> <tr><td>178,38</td><td>744,91</td><td>1,52</td><td>1003,76</td></tr> <tr><td>190,3</td><td>1489,52</td><td>7,03</td><td>10051</td></tr> <tr><td>200,63</td><td>2806,78</td><td>8,48</td><td>22849,1</td></tr> <tr><td>219,74</td><td>7875,99</td><td>9,34</td><td>59231,6</td></tr> <tr><td>313,61</td><td>127,21</td><td>25,88</td><td>2585,03</td></tr> <tr><td>416,93</td><td>1285,65</td><td>24,88</td><td>25420,7</td></tr> <tr><td>483,12</td><td>262,81</td><td>9,91</td><td>2086,24</td></tr> <tr><td>504,08</td><td>1170,61</td><td>22,75</td><td>21388,8</td></tr> <tr><td>531,63</td><td>1661,03</td><td>20,04</td><td>26827</td></tr> <tr><td>586,76</td><td>424,51</td><td>28,04</td><td>9660,55</td></tr> <tr><td>627,55</td><td>2733,32</td><td>35,64</td><td>79453,2</td></tr> </tbody> </table>	sommet	Amplitude	largeur	Surface	100,51	4704,43	6,17	22233,9	11,65	8416,24	13,29	102392	128,04	5267,3	8,22	40656,7	150,3	4476,77	7,95	33774	178,38	744,91	1,52	1003,76	190,3	1489,52	7,03	10051	200,63	2806,78	8,48	22849,1	219,74	7875,99	9,34	59231,6	313,61	127,21	25,88	2585,03	416,93	1285,65	24,88	25420,7	483,12	262,81	9,91	2086,24	504,08	1170,61	22,75	21388,8	531,63	1661,03	20,04	26827	586,76	424,51	28,04	9660,55	627,55	2733,32	35,64	79453,2
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### CuAg1Au3

**Recette :**

**Rokusho 4 :**

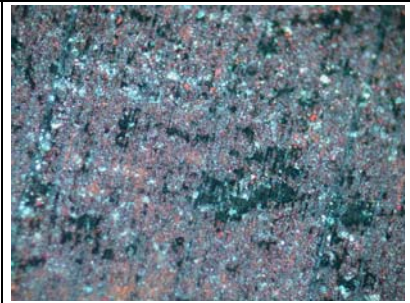
- 30 g acétate de cuivre
- 30 g sulfate de cuivre
- 90 cl acide acétique dilué 5%



MO BF

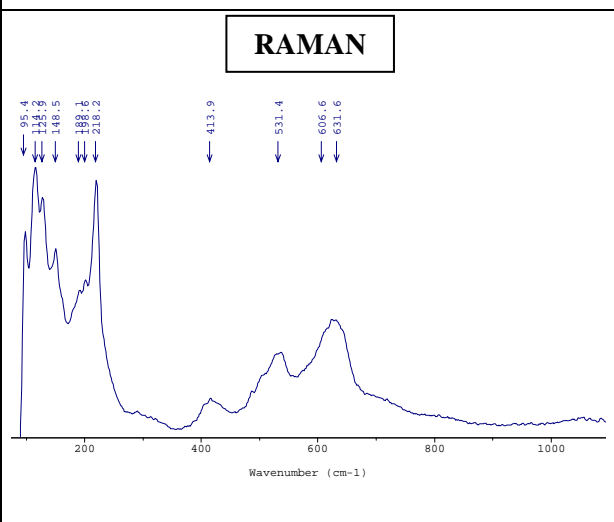
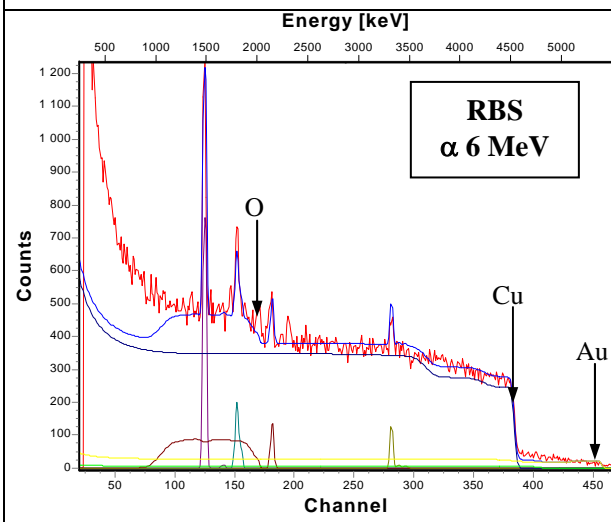
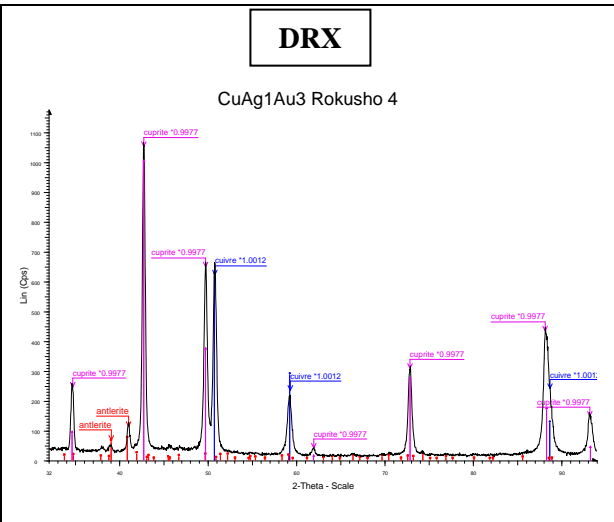
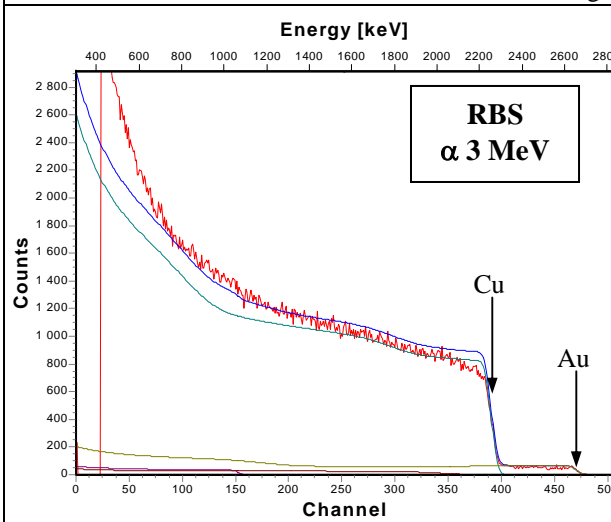


MO DF



MO P.LA

PIXE: Cu95% Ag1.1% Au3.1% S0.8%



## Description

<b>Composition Couche</b>																																																																		
Cu45 O25 Au0.5 Porosité + rugosité Cu/Au=90	6000.10 <sup>15</sup> At/cm <sup>2</sup>	<b>Diffraction X</b>  Cuprite Décalée *0.9977  Cuivre Décalé *1.0012  <b>Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes</b>  <b>CuAg1Au3 R4 - cm<sup>-1</sup></b> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>sommet</th> <th>Amplitude</th> <th>largeur</th> <th>Surface</th> </tr> </thead> <tbody> <tr><td>95,59</td><td>725,18</td><td>5,49</td><td>3296,95</td></tr> <tr><td>113,12</td><td>1511,37</td><td>13,49</td><td>17671,7</td></tr> <tr><td>127,32</td><td>1073,32</td><td>13,14</td><td>13059,7</td></tr> <tr><td>148,89</td><td>611,54</td><td>13,05</td><td>6146,07</td></tr> <tr><td>178,91</td><td>96,54</td><td>5,81</td><td>433,73</td></tr> <tr><td>189,61</td><td>243,23</td><td>9,33</td><td>1755,77</td></tr> <tr><td>199,79</td><td>356,82</td><td>9,68</td><td>2978,96</td></tr> <tr><td>217,78</td><td>1458,18</td><td>11,81</td><td>14204,6</td></tr> <tr><td>412,95</td><td>213,65</td><td>29,92</td><td>5078,47</td></tr> <tr><td>485,11</td><td>111,08</td><td>8,64</td><td>879,85</td></tr> <tr><td>502,33</td><td>185,12</td><td>14,38</td><td>2137,95</td></tr> <tr><td>527,76</td><td>372,47</td><td>23,25</td><td>6976,9</td></tr> <tr><td>599,1</td><td>664,51</td><td>29,04</td><td>15733,1</td></tr> <tr><td>625,34</td><td>223,25</td><td>28,56</td><td>5181,13</td></tr> <tr><td>647,97</td><td>285,39</td><td>19,68</td><td>4592,4</td></tr> </tbody> </table>	sommet	Amplitude	largeur	Surface	95,59	725,18	5,49	3296,95	113,12	1511,37	13,49	17671,7	127,32	1073,32	13,14	13059,7	148,89	611,54	13,05	6146,07	178,91	96,54	5,81	433,73	189,61	243,23	9,33	1755,77	199,79	356,82	9,68	2978,96	217,78	1458,18	11,81	14204,6	412,95	213,65	29,92	5078,47	485,11	111,08	8,64	879,85	502,33	185,12	14,38	2137,95	527,76	372,47	23,25	6976,9	599,1	664,51	29,04	15733,1	625,34	223,25	28,56	5181,13	647,97	285,39	19,68	4592,4
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647,97	285,39	19,68	4592,4																																																															
Cu66 Ag0.6 Au0.6 O33 Cu/Au=110 Cu/Ag=110	8000.10 <sup>15</sup> At/cm <sup>2</sup>																																																																	
Cu98.4 Au1 Ag0.6 Cu/Au=98.4 Cu/Ag=164	Substrat																																																																	

# CuSn3Ag2Au6

## Recette :

### Rokusho 4 :

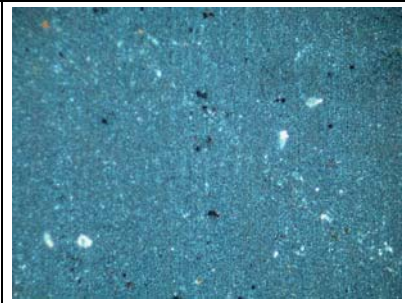
- 30 g acétate de cuivre
- 30 g sulfate de cuivre
- 90 cl acide acétique dilué 5%



MO BF

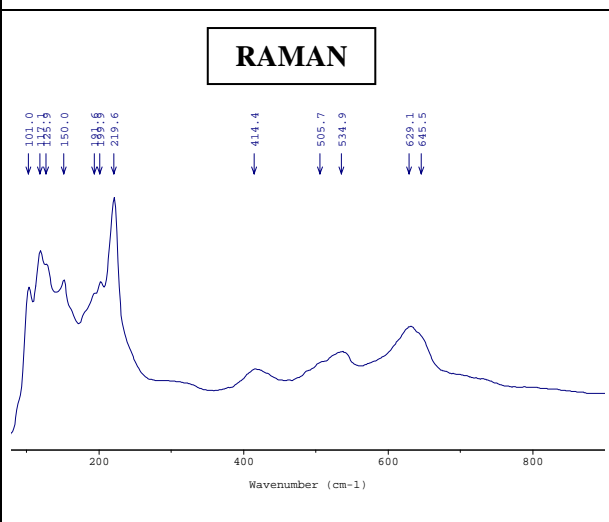
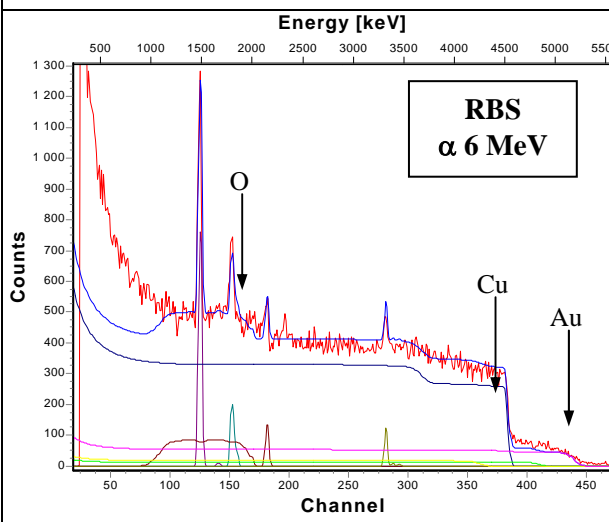
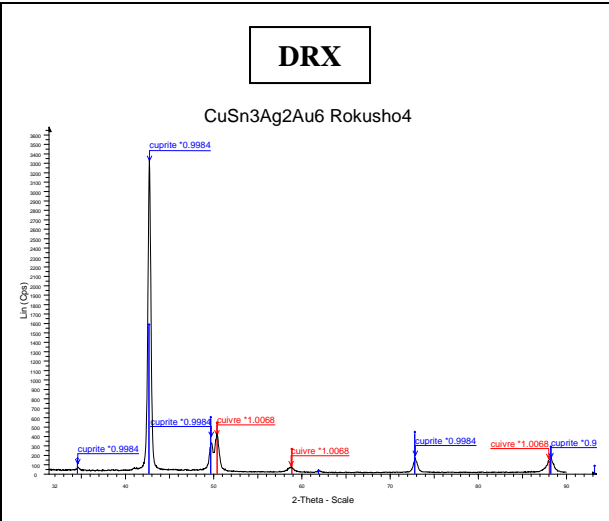
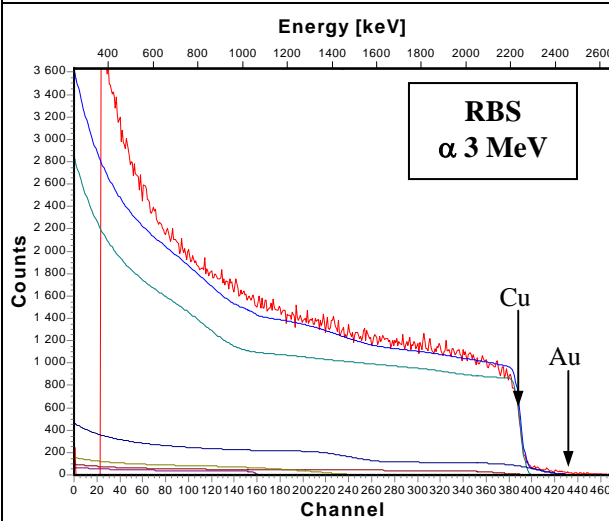


MO DF



MO PL A

PIXE: Cu87.5% Sn3.3% Ag2.6% Au6.3% S0.3%



<b>Description</b>																																																																							
<b>Composition Couche</b>																																																																							
Cu50 O25 Ag0 Au0 Porosité +rugosité	4000.10 <sup>15</sup> At/cm <sup>2</sup>	<b>Diffraction X</b>  Cuprite Décalée *0.9984  Cuivre Décalé *1.0068  <b>Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes</b>  <b>CuSn3Ag2Au6 R4</b> cm <sup>-1</sup> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>sommet</th> <th>Amplitude</th> <th>largeur</th> <th>Surface</th> </tr> </thead> <tbody> <tr><td>100,82</td><td>2169,83</td><td>9,37</td><td>16247,5</td></tr> <tr><td>116,23</td><td>2482,35</td><td>10,7</td><td>22522</td></tr> <tr><td>126,96</td><td>2595,76</td><td>19,93</td><td>40600,6</td></tr> <tr><td>149,91</td><td>1872,58</td><td>13,27</td><td>19378,7</td></tr> <tr><td>180,35</td><td>220,88</td><td>5,69</td><td>973,29</td></tr> <tr><td>190,52</td><td>634,63</td><td>7,8</td><td>3829,69</td></tr> <tr><td>200,69</td><td>1396,92</td><td>12,28</td><td>15537,1</td></tr> <tr><td>219,46</td><td>5076,62</td><td>12,04</td><td>49019,6</td></tr> <tr><td>311,91</td><td>113,34</td><td>25,47</td><td>2265,6</td></tr> <tr><td>415,24</td><td>656,29</td><td>28,12</td><td>14598,3</td></tr> <tr><td>486,05</td><td>165,34</td><td>8,03</td><td>1064,53</td></tr> <tr><td>504,79</td><td>466,19</td><td>19,58</td><td>7333,75</td></tr> <tr><td>531,64</td><td>844,82</td><td>22,3</td><td>15183,2</td></tr> <tr><td>599,98</td><td>302,92</td><td>23,28</td><td>5714,49</td></tr> <tr><td>629,99</td><td>1750,44</td><td>30,91</td><td>44257,6</td></tr> <tr><td>650,13</td><td>386,71</td><td>17,16</td><td>5424,65</td></tr> </tbody> </table>		sommet	Amplitude	largeur	Surface	100,82	2169,83	9,37	16247,5	116,23	2482,35	10,7	22522	126,96	2595,76	19,93	40600,6	149,91	1872,58	13,27	19378,7	180,35	220,88	5,69	973,29	190,52	634,63	7,8	3829,69	200,69	1396,92	12,28	15537,1	219,46	5076,62	12,04	49019,6	311,91	113,34	25,47	2265,6	415,24	656,29	28,12	14598,3	486,05	165,34	8,03	1064,53	504,79	466,19	19,58	7333,75	531,64	844,82	22,3	15183,2	599,98	302,92	23,28	5714,49	629,99	1750,44	30,91	44257,6	650,13	386,71	17,16	5424,65
sommet	Amplitude			largeur	Surface																																																																		
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650,13	386,71	17,16	5424,65																																																																				
Cu65 Ag 1 Au1 O33 Cu/Au=65 Cu/Ag=65	7000.10 <sup>15</sup> At/cm <sup>2</sup>																																																																						
Cu64 Sn1 Ag 1 Au1.8 O32.2 Cu/Au=35.5 Cu/Ag=64	2000.10 <sup>15</sup> At/cm <sup>2</sup>																																																																						
Cu94.5 Ag1.2 Au2 Sn1.7 Cu/Au=315 Cu/Ag=52	Substrat																																																																						
<b>Analyse XPS:</b> Or et Argent non visibles en surface ni même après une abrasion de 40 min (env. 400nm)																																																																							

# CuSn6Ag3Au1

**Recette :**

**Rokusho 4 :**

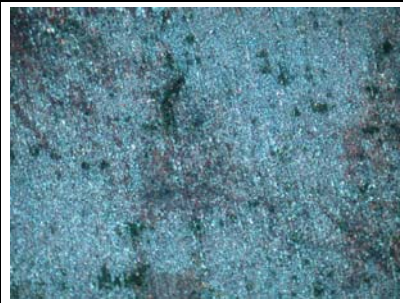
30 g acétate de cuivre  
 30 g sulfate de cuivre  
 90 cl acide acétique dilué 5%



MO BF

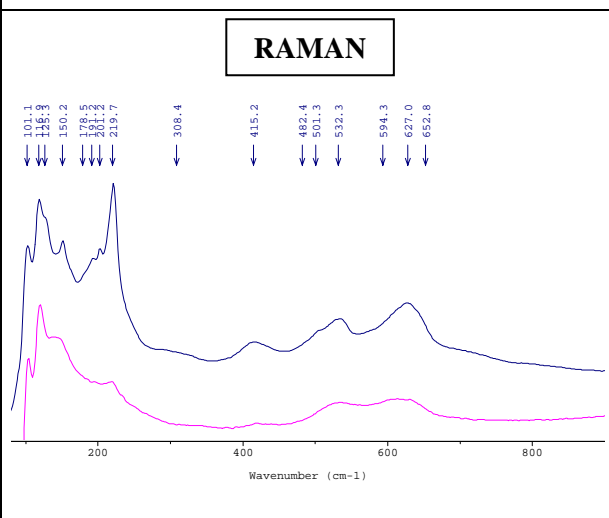
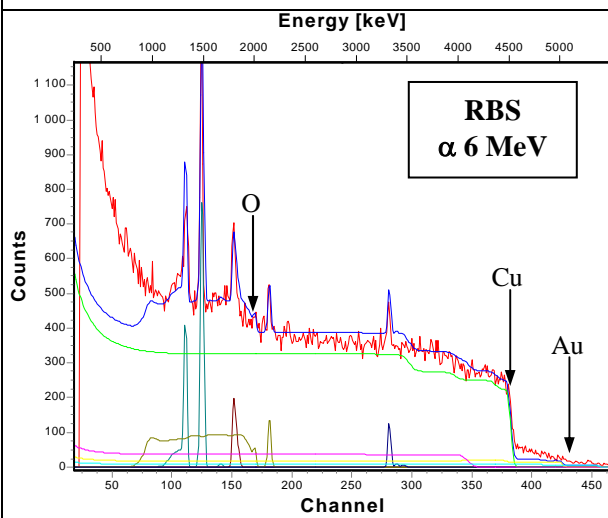
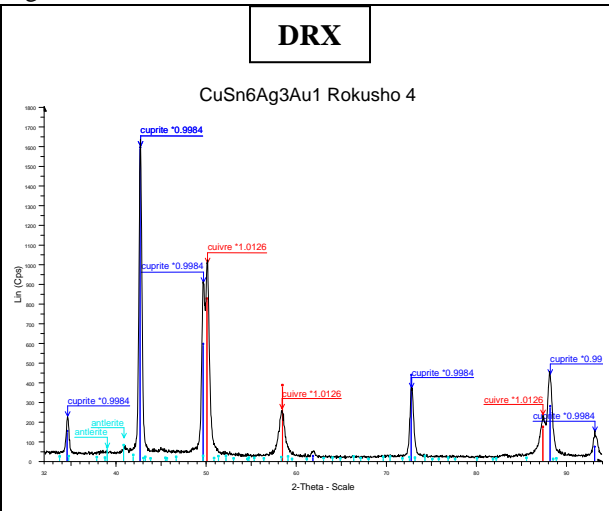
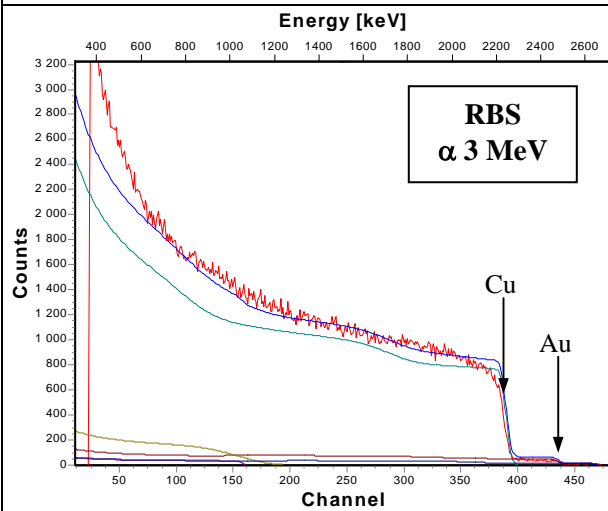


MO DF



MO PLA

PIXE: Cu88.7% Sn6.1% Ag3.6% Au1.1% S0.3%



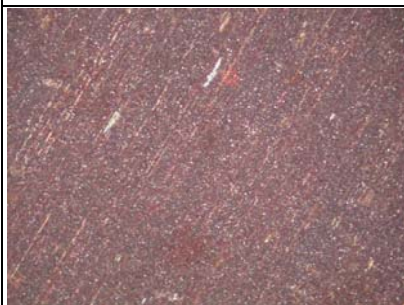
Description																																																																			
Composition Couche																																																																			
Cu40 O25 Ag 1 Au0.1 Porosité +rugosité Cu/Au=400 Cu/Ag=40	7000.10 <sup>15</sup> At/cm <sup>2</sup>	<b>Diffraction X</b>																																																																	
Cu66 Ag 1.8 Au0.3 O31.9 Cu/Au=220 Cu/Ag=36.6	7500.10 <sup>15</sup> At/cm <sup>2</sup>	Cuprite Décalée *0.9984																																																																	
Cu94.5 Ag1.8 Au0.3 Sn 3.4 Cu/Au=315 Cu/Ag=52	Substrat	Cuivre Décalé *1.0126																																																																	
		<b>Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes</b>																																																																	
		<b>CuSn6Ag3Au1 R4</b>																																																																	
		point2 cm <sup>-1</sup>																																																																	
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## CuSn5Pb5

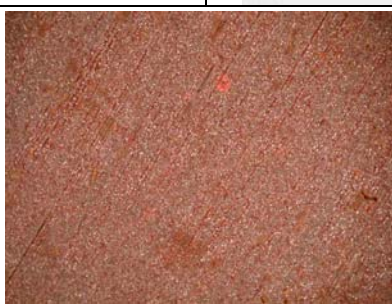
### Recette :

#### Rokusho 4 :

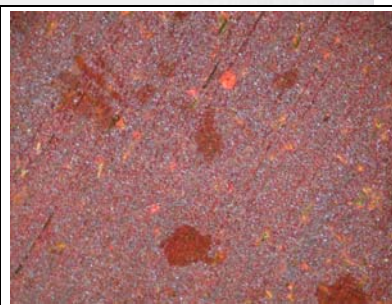
30 g acétate de cuivre  
 30 g sulfate de cuivre  
 90cl acide acétique dilué 5%



MO BF

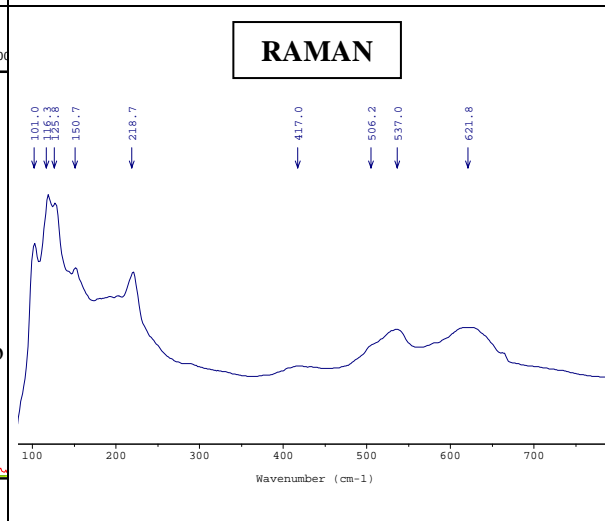
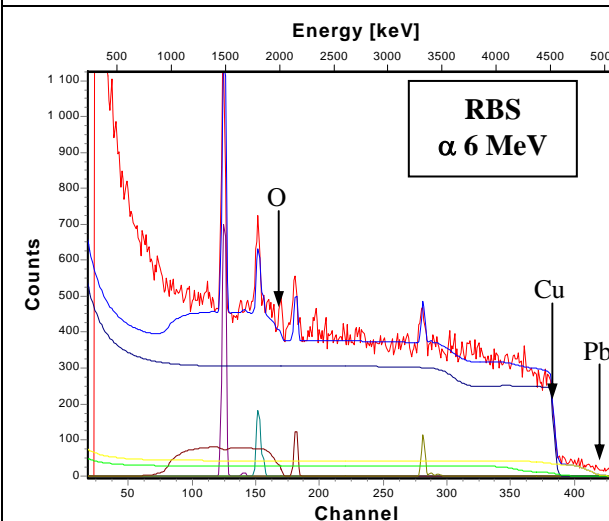
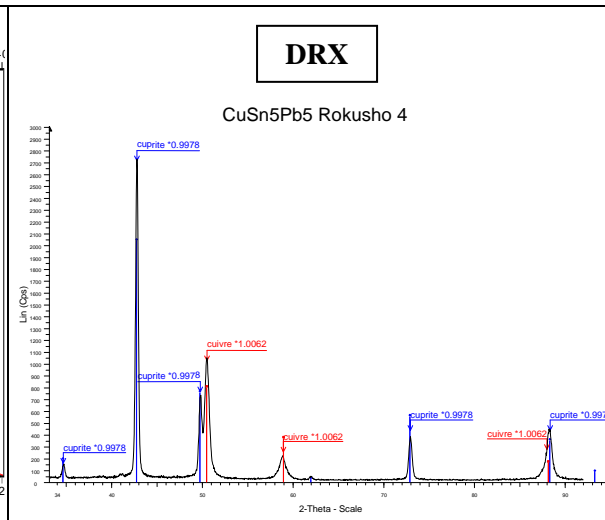
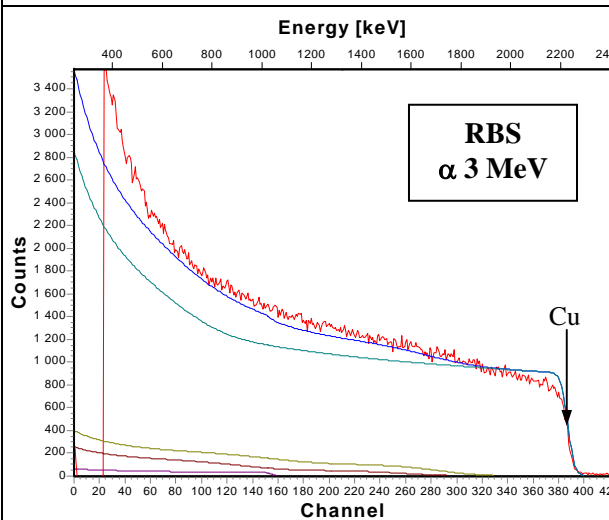


MO DF



MO P.L.A

PIXE: Cu94.4% Sn4.6% Pb0.9% S0.1%



## Description

Composition Couche																																																																	
Cu60 O30 Porosité +rugosité	9000.10 <sup>15</sup> At/cm <sup>2</sup>	<b>Diffraction X</b>  Cuprite Décalée *0.9978  Cuivre Décalé *1.0062  <b>Dépouillement RAMAN : cuprite seule, avec reconstruction des bandes complexes</b>  <b>CuSn5Pb5 R4</b> cm <sup>-1</sup> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>sommet</th> <th>Amplitude</th> <th>largeur</th> <th>Surface</th> </tr> </thead> <tbody> <tr><td>101,18</td><td>1483,45</td><td>3,86</td><td>4385,51</td></tr> <tr><td>117,27</td><td>3558,9</td><td>9,52</td><td>30758,6</td></tr> <tr><td>125,82</td><td>3963,29</td><td>13,79</td><td>47625,9</td></tr> <tr><td>150,19</td><td>110,34</td><td>10,99</td><td>9400,9</td></tr> <tr><td>190,65</td><td>127,46</td><td>7,05</td><td>695,43</td></tr> <tr><td>201,21</td><td>230,04</td><td>10,68</td><td>2351,46</td></tr> <tr><td>219,3</td><td>1571,92</td><td>10,17</td><td>12415,3</td></tr> <tr><td>416,96</td><td>337,46</td><td>28,64</td><td>7683,15</td></tr> <tr><td>485,85</td><td>126,75</td><td>18,13</td><td>1842,12</td></tr> <tr><td>508,23</td><td>810,62</td><td>19,92</td><td>12974</td></tr> <tr><td>532,34</td><td>1489,1</td><td>21,77</td><td>26131,9</td></tr> <tr><td>585,73</td><td>230,93</td><td>23,63</td><td>4427,64</td></tr> <tr><td>621,9</td><td>1520,48</td><td>35,53</td><td>44026,2</td></tr> <tr><td>643</td><td>173,01</td><td>11,28</td><td>1594,76</td></tr> </tbody> </table>				sommet	Amplitude	largeur	Surface	101,18	1483,45	3,86	4385,51	117,27	3558,9	9,52	30758,6	125,82	3963,29	13,79	47625,9	150,19	110,34	10,99	9400,9	190,65	127,46	7,05	695,43	201,21	230,04	10,68	2351,46	219,3	1571,92	10,17	12415,3	416,96	337,46	28,64	7683,15	485,85	126,75	18,13	1842,12	508,23	810,62	19,92	12974	532,34	1489,1	21,77	26131,9	585,73	230,93	23,63	4427,64	621,9	1520,48	35,53	44026,2	643	173,01	11,28	1594,76
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Cu67.2 Sn1 Pb0.8 O31	5000.10 <sup>15</sup> At/cm <sup>2</sup>																																																																
Cu95.6 Sn2.8 Pb1.6 Cu/Au=315 Cu/Ag=52	Substrat																																																																

## CuSn3Ag2Au6

**Recette :**

**Murakami :**

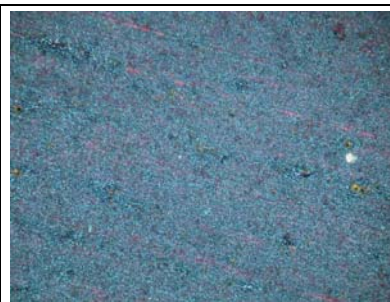
- 1,9 g acétate de cuivre
- 1,2 g sulfate de cuivre
- 0,1 g alun
- 1 l eau



MO BF

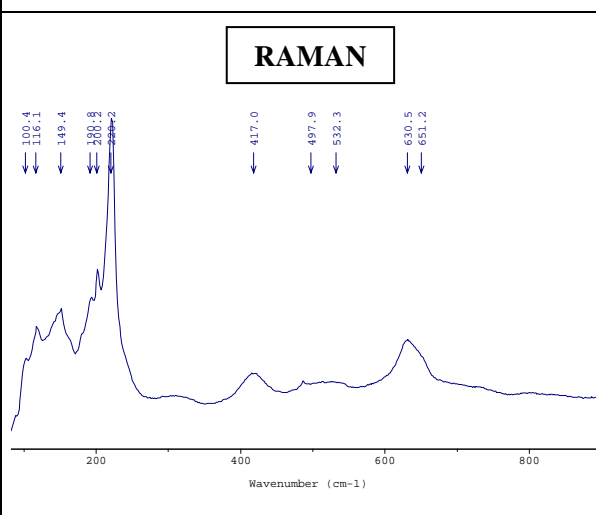
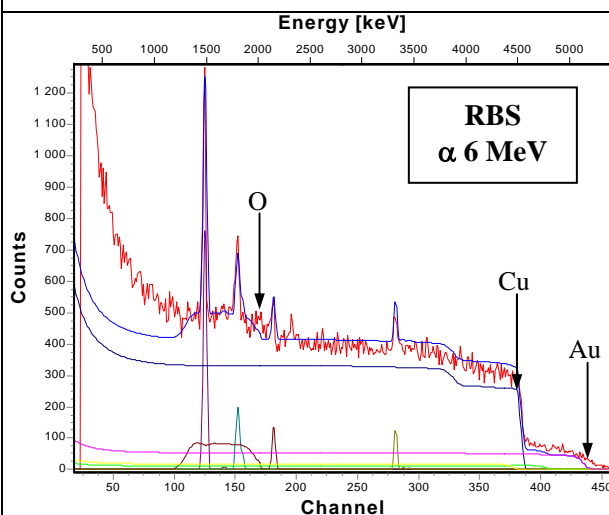
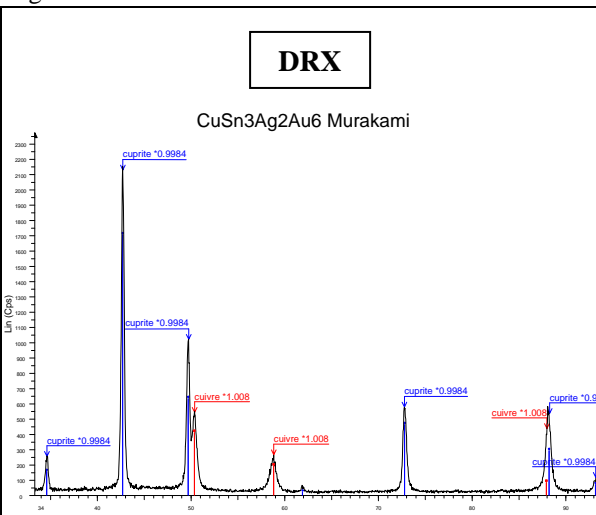
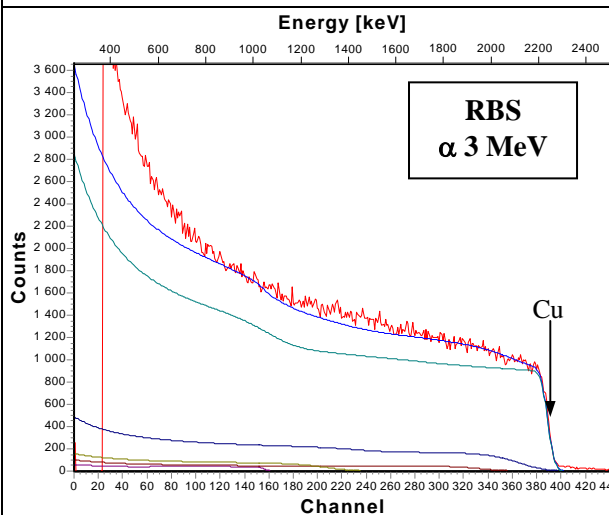


MO DF



MO P.LA

PIXE: Cu89.1% Ag2.4% Au5.7%



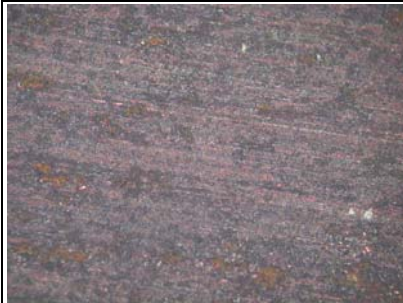
<b>Description</b>		
<b>Composition Couche</b>		
Cu63 O37 Ag0 Au0 +rugosité	5000.10 <sup>15</sup> At/cm <sup>2</sup>	<b>Diffraction X</b>
Cu68 Ag1.2 Au1.5 O29 Cu/Au=45 Cu/Ag=57	5500.10 <sup>15</sup> At/cm <sup>2</sup>	Cuprite Décalée *0.9984
Cu94.5 Ag1.2 Au2 Sn1.7 Cu/Au=315 Cu/Ag=52	Substrat	Cuivre Décalé *1.008

# CuSn6Ag3Au1

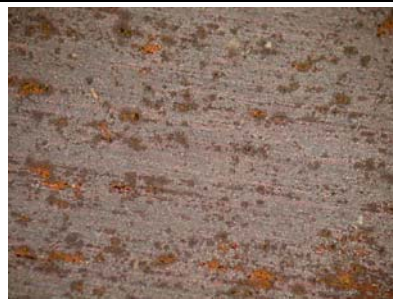
**Recette :**

**Murakami :**

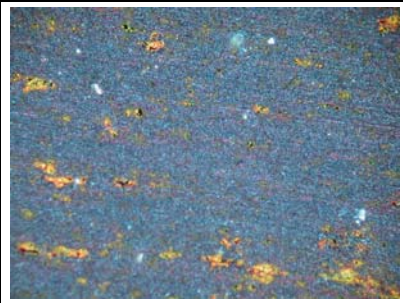
- 1,9 g acétate de cuivre
- 1,2 g sulfate de cuivre
- 0,1 g alun
- 1 l eau



MO BF

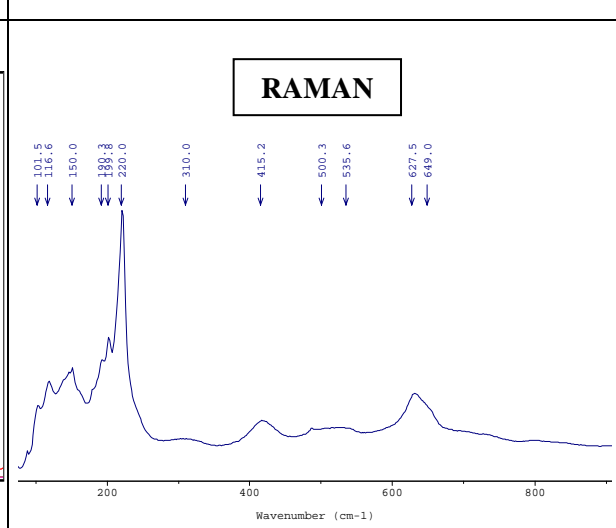
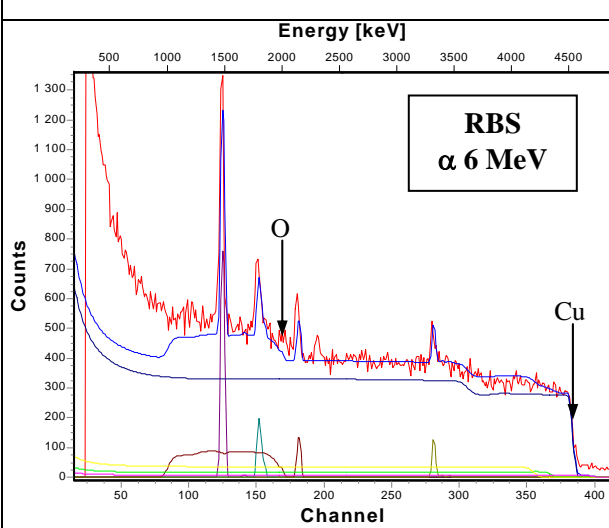
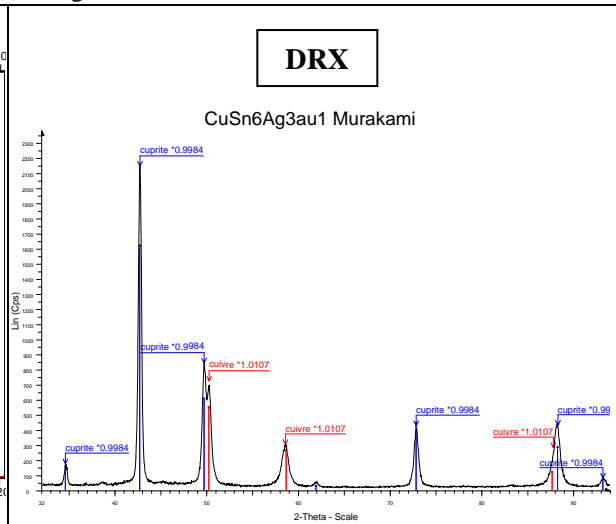
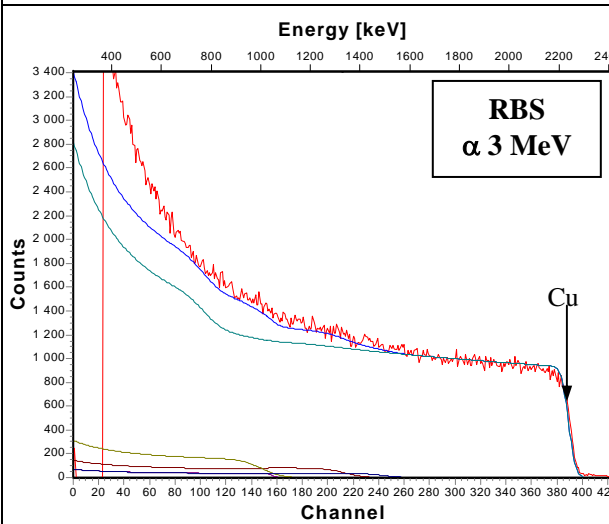


MO DF



MO PLA

PIXE: Cu89.1% Sn5.9% Ag3.2% Au0.9%



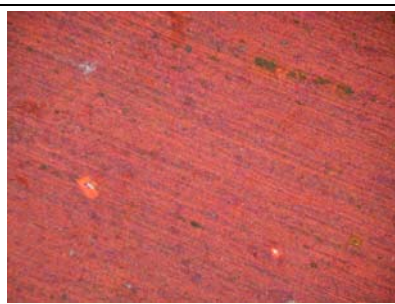
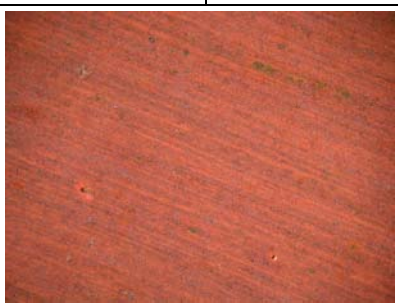
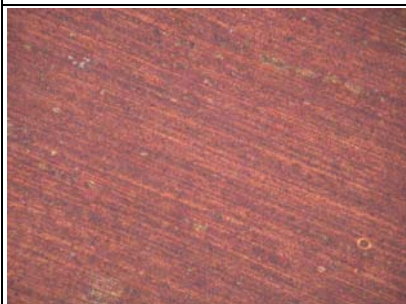
<b>Description</b>		
<b>Composition Couche</b>		
Cu67 O33 Ag0 Au0 rugosité	10000.10 <sup>15</sup> At/cm <sup>2</sup>	<b>Diffraction X</b>
Cu67 Ag 1.8 Au0.3 O30.9 Cu/Au=220 Cu/Ag=36.6	3000.10 <sup>15</sup> At/cm <sup>2</sup>	Cuprite Décalée *0.9984
Cu94.5 Ag1.8 Au0.3 Sn 3.4 Cu/Au=315 Cu/Ag=52	Substrat	Cuivre Décalé *1.0107

## CuSn5Pb5

**Recette :**

**Murakami :**

- 1,9 g acétate de cuivre
- 1,2 g sulfate de cuivre
- 0,1 g alun
- 1 l eau

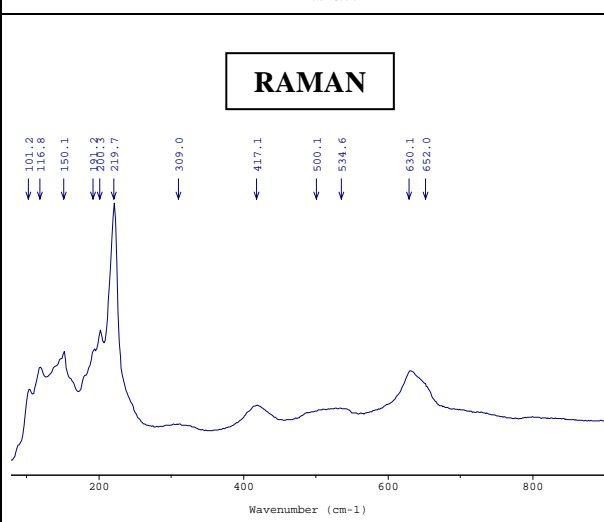
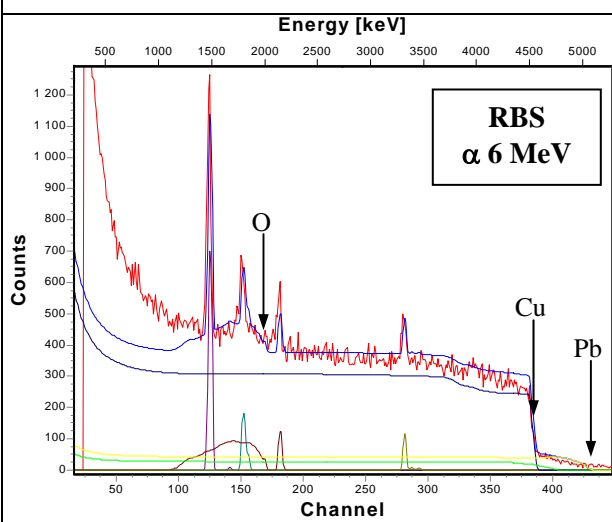
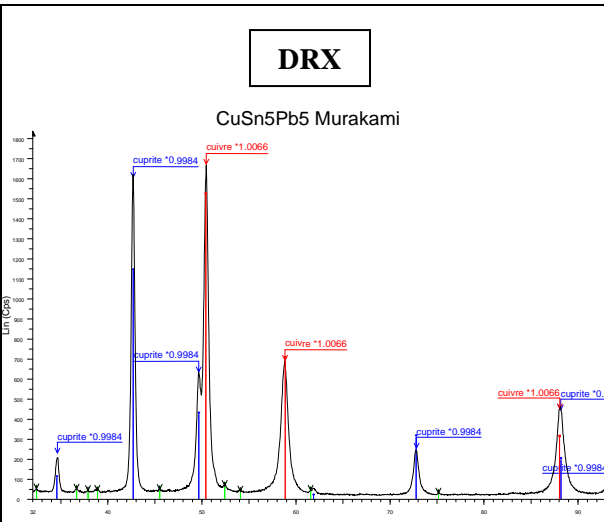
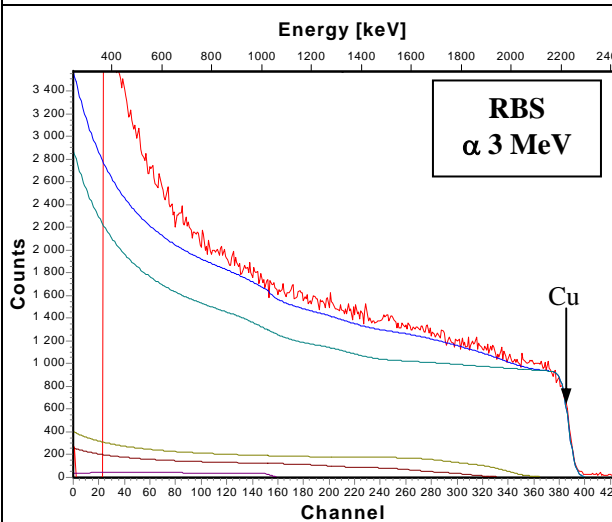


MO BF

MO DF

MO PLA

PIXE: Cu94% Sn3.4% Pb2.6%



<b>Description</b>		
<b>Composition Couche</b>		
Cu66 O34 rugosité	6000.10 <sup>15</sup> At/cm <sup>2</sup>	<b>Diffraction X</b>  Cuprite Décalée *0.9984  Cuivre Décalé *1.0066
Cu68 Sn1 Pb1 O30	2000.10 <sup>15</sup> At/cm <sup>2</sup>	
Cu78 Sn2 Pb1.5 O18.5	2500.10 <sup>15</sup> At/cm <sup>2</sup>	
Cu95.6 Sn2.8 Pb1.6 Cu/Au=315 Cu/Ag=52	Substrat	