In memoriam tribute to Richard Penelle (1936-2012)

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Abstract. The present paper is dedicated to Richard Penelle who was an internationally recognized texture specialist. The first part quickly describes his scientific carrier and the second one presents his last paper as first author and his last research projects on the Invar and 1050 aluminium recrystallization, respectively. Most of his last studies were linked to the understanding of the Cube texture development in fcc materials.

1. The scientific carrier of Richard Penelle
Richard Penelle was an immutable figure of texture specialist at all national, European and international texture conferences, except the last ICOTOM 16 in December 2011, Mumbai. At that time, Richard was already severely ill of a disease which took him away just before Christmas 2012.

After a solid scientific education at the historical Sorbonne university, Richard Penelle prepared his thesis at the Centre de Recherches Métallurgiques (CRM) of the Ecole des Mines de Paris, under the supervision of Professor Paul Lacombe (see figure 1).

Figure 1: Meeting at the "Maison de la Chimie", Paris, September 1989. From the left to the right: Dr. T. Okada, Prof. J. Philibert, Prof. P. Lacombe, Dr. R. Penelle and Dr. M. Aucouturier (Photo: courtesy of Prof. J. Philibert)

The title of his thesis, defended in 1967, was "Relations between deformation mechanisms in rolled iron single crystals and their deformation and primary recrystallization textures". In 1983, Professor Lacombe received the sword of Academician with the specialty metallurgy, a specialty which is only exceptionally honored by the Academy of Sciences. All along his career at the French CNRS, Richard Penelle continued to keep metallurgy as a common thread in his wide ranging research on metallic materials from iron and low alloy steels, to alloys of iron and nickel, and even to zirconium and titanium and their alloys. While conducting a fundamental scientific approach, he became also interested in the technological aspects involved, e.g. his contribution to deep drawing.

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Even after the scientific meeting held in 2009 in his honor at Orsay at the occasion of his "official" retirement as a Research Director of the CNRS, Richard Penelle continued his scientific activity and loved to share his knowledge with the younger scientists of his former small research group, researchers who pursue studies on the topics he had initiated.

Richard Penelle had also an intense international cooperation with many foreign countries during his entire career. When he fell ill, he was just returned from his last mission to Brazil, where he gave a series of lectures at the Mining School of Ouro Preto.

Despite his dense scientific activity, Richard also enjoyed an intense and harmonious family life with his wife, Gabrielle, who was his faithful companion and stood on his side at most of his missions, notably the ICOTOM conferences. She offered him three daughters Anne, Isabelle and Aude, who gave him five grandchildren and his family accompanied him to the end.

2. Some last scientific achievements of Richard Penelle

From Web of Science™, it appears that Richard Penelle has published his last paper as a first author in 2010 on recrystallization of the Fe-36%Ni (Invar) alloy [1]. However, until the end, he continued to supervise research works of the Orsay team. The last one concerned the recrystallization study of 1050 aluminium alloy. Richard Penelle has co-published two last papers in international journals in 2012 on this subject [2,3].

Let us note that in 2012 he has also published three other papers in the Proceedings of ICOTOM 16 (Bombay, 2011) [4-6] and a book chapter on the influence of textures on the physical properties [7].

In the present paper dedicated to Richard Penelle, instead of presenting broadly the results obtained during his long scientific carrier, it was decided to select some results of his last publication [1] and last research project [2-4]. These two studies are linked to the understanding of the Cube texture development during recrystallization of cold rolled sheets, of Invar and 1050 aluminium alloy.

2.1. Primary recrystallization of Invar

The French German “Texture and Anisotropy” symposium, organized by Claude Esling and Richard Penelle, for the SF2M-DGM societies on April 3rd 2009, was preceded by a scientific day to honour Richard Penelle at the occasion of his “official” retirement. During this day, organized by Thierry Baudin and Claude Esling, Richard Penelle presented a communication entitled “On primary recrystallization of Invar”. The corresponding paper was then published in a special issue of Advanced Engineering Materials. It was his last paper as first author.

This paper was a short review of results obtained with his team on recrystallization of the Fe-36%Ni (Invar) alloy. The Fe-36%Ni austenitic alloy is used in electronic devices because of its low thermal expansion coefficient and its good magnetic properties [8]. For that purpose it is beneficial to have a high fraction of Cube orientation. These works have started with the two postdoctoral years of Stefan Zaefferer (10-1995-10-1997), and then with the PhD theses of Frank Julliard (2001) [9] and Sorphal Chhann (2007) [10] plus some additional contributions of his close collaborators.

The Cube orientation nucleation, notably its development after high deformation by cold rolling and subsequent annealing, was discussed from multiscale analyses based on TEM, SEM/EBSD, X-ray and neutron diffraction measurements. It was explained why the Cube texture sharpness increased with the deformation amount.

These experimental observations were also coupled with a quite original Monte-Carlo simulation, since the calculation was performed from a TEM crystallographic orientation map and stored energy values estimated by neutron diffraction for all the components of the deformation texture [11].

From these studies, which were also completed by additional work on Fe-Ni alloys supervised by Richard Penelle (see [12] for example), some results have been highlighted.

After 95% cold rolling, the Copper texture is developed. It is composed of the C {112}<111>, B {110}<112> and S {123}<634> components [13]. In addition, a minor Cube {001}<100> orientation is also observed. On the contrary, after complete recrystallization (60 min at
600°C), a sharp Cube texture is observed (about 80%). At the same time, its twin \{122\}<221> orientation appears (about 10%) and a random part remains (about 10%). The Cube orientation nucleates preferentially because of its cellular dislocation substructure which presents a low stored energy. On the contrary, the other deformation texture components have a lamellar substructure with a high stored energy. In fact, the most important parameter is the stored energy difference between the Cube and the non-Cube orientations which increases with the deformation amount and explains why the Cube orientation is the main recrystallization component for the highest cold rolling reduction. For low reductions (<70%), this parameter is quite low and all the deformation texture components can nucleate. For very low reduction, because of twinning, the recrystallization texture tends to become random [1]. So, after dislocation cell growth or coalescence to attain a critical size, a Cube subgrain (named A in figure 2) adjacent to a grain boundary (high misorientation), can grow by Strain Induced Boundary Migration (SIBM) as shown in figure 2.

Figure 2: TEM orientation map \{hkl\} distribution) showing a Cube band in a 95% cold rolled and 2 min. at 600°C annealed sample [9].

This research work was initiated by Richard Penelle in the years 1994-1995 but a lot of work still remains to improve the Cube texture development. Indeed, despite of a better understanding of the metallurgical evolution, the Orsay team currently continues to work on this topic and particularly on the Fe-48%Ni recrystallization but for other applications such as photovoltaic cells. In this case, it is necessary to develop 100% of Cube texture with a low dispersion (lower than 5°) [14] to lead to an epitaxial deposition of silicon on the Fe-Ni substrate. In order to succeed, the cold rolling reduction is increased up to 99% and the thermal treatment must be optimized as well as the material chemical composition. As an example, figure 3 shows the adverse effect of niobium amount on the Cube fraction.

Figure 3: EBSD maps of Fe-48%Ni alloy with (a) 0%, (b) 0.035% and (c) 0.050% of Nb (weight %) - \{hkl\} distribution) [14]

This figure shows that the Cube fraction decreases (99.9 %, 98.8 % and 97.2 %, respectively, with a 10° misorientation from the ideal Cube orientation) when the percentage of niobium increases because niobium is a recrystallization inhibitor. On the contrary, it was shown that sulphur favours the Cube development through MnS precipitation. Indeed, they are the origin of hardening that increases the stored energy gap between Cube and the other components. Thus, this difference favours the Cube development [14].
2.2. Primary recrystallization of 1050 aluminium alloy

In the year 2010, Richard Penelle and his close colleague Anne-Laure Helbert started to work on the formation and the development of a strong Cube recrystallization texture in aluminium of commercial purity, in collaboration with François Boutin, previously research director of the Alcan society. Wei Wang worked on this topic in 2010 as part of his master thesis [15] before starting a PhD project in the same research team on recrystallization of NiCrW alloys [16].

The manufacture of electrolytic capacitors requires the development of a very pronounced Cube texture. So, Boutin [17] proposed an industrial process to develop a predominant Cube texture in high purity aluminium. However in the case of commercial purity aluminium of low cost, the Cube fraction remains lower than about 30%. Knowing that silicon and iron contents in solid solution slow down the Cube texture development, the first idea was to introduce a pre-annealing for 5h at 300°C to precipitate Si and Fe aluminides to limit solute dragging effect in the 1050 aluminium of commercial purity. Moreover, since the energy stored during deformation provides the driving force for recrystallization, the authors proposed to introduce an additional 10% cold rolling before the final annealing after a partial recrystallization of the alloy at moderate temperature (200°C). Because of the geometry of the slip systems, the Cube grains are less affected by strain hardening than the non-Cube grains. Thus, the stored energy difference is increased and the Cube grain nucleation is favoured. Under these conditions, the optimized thermo-mechanical process, described in figure 4, allows to develop about 65% of Cube grains, in contrast to 30% using a more traditional route.

![Figure 4: Processing route established to develop 65% of Cube texture after final recrystallization [2-4].](image)

Obviously, this fraction remains low with regard to that developed in high purity aluminium. However, this result is encouraging, but to still increase the Cube fraction, the nucleation and growth mechanisms of Cube grains have been studied [2-4].

To illustrate the necessity to introduce a pre-annealing for 5h at 300°C, figure 5 shows that this annealing allows to form Si and Fe aluminide precipitates. According to the study of Zhang et al. [18], most of them are AlFeSi and FeSiAl5 intermetallic compounds. This pre-annealing allows doubling the Cube fraction [2-4].

![Figure 5: TEM micrographs of the 1050 aluminium alloy (a) in the as-received state without precipitates and (b) after pre-annealing, with precipitates [2-4].](image)
As observed in the Invar alloy, the dislocation substructure in Cube grains is composed of equiaxed dislocation cells. On the contrary, the other deformation texture components present a lamellar or an irregular substructure. Moreover, it has been observed that the Cube grains recover quickly compared to the other components [19-21]. Thus, due to its dislocation cell shape and the stored energy difference, the Cube nucleation is favoured. The 10% cold rolling before the final annealing allows to further increase this stored energy difference and then favours the Cube grain growth by strain induced boundary migration (SIBM) [2-4]. Besides, a second recrystallization mechanism is observed. During annealing, the substructure of polygonized Cube grains is removed by in-situ recrystallization (or generalized recovery). As an example, figure 6 shows the sub-structure evolution in a Cube grain observed during in-situ annealing in a FEG-SEM from room temperature up to 450°C with a heating rate of 55°C/h on a sample partially recrystallized at 200°C and then 10% cold-rolled (see figure 4). This mechanism promotes the development of the Cube orientation in the texture.

![Figure 6: EBSD maps showing the sub-structure evolution of a Cube grain at (a) 290°C, (b) 310°C and (c) 350°C observed during in-situ annealing in a FEG-SEM (from [2-4]).](image)

Apart from this mechanism, that clearly shows that the Cube development is linked to the Cube dislocation cell morphology, it was also evidenced that the stored energy difference between Cube grains (low energy) and non-Cube grains (high energy) promotes the Cube development, as well as in the FeNi alloys. However, even if the Cube nucleation and growth mechanisms are now quite well understood, it still remains to increase the Cube volume fraction by optimizing the thermo-mechanical process.

### 3. Conclusion

During his long carrier mainly dedicated to the understanding of the correlation between microstructure, texture and physical properties, Richard Penelle has contributed to a better understanding of the deformation, recovery, primary recrystallization and (normal or abnormal) grain growth mechanisms in different metallic or geological materials of various crystal structures.

In this paper, as an example, the understanding of the Cube texture development was particularly discussed in Invar and 1050 aluminium alloys. The Cube texture fraction was increased notably thanks to Richard Penelle’s research. However, it still remains a lot of work to do for the young co-workers to carry on the studies initiated by him.

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